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TECHNOLOGY
DEPARTMENT

Corrosion

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OFFICIAL PUBLICATION
NATIONAL ASSOCIATION
OF CORROSION ENGINEERS

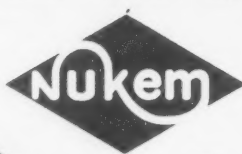
JULY

1961

Also: Classified New Product News About
Corrosion Control Products, Services

Complete Contents on Pages 4 and 5

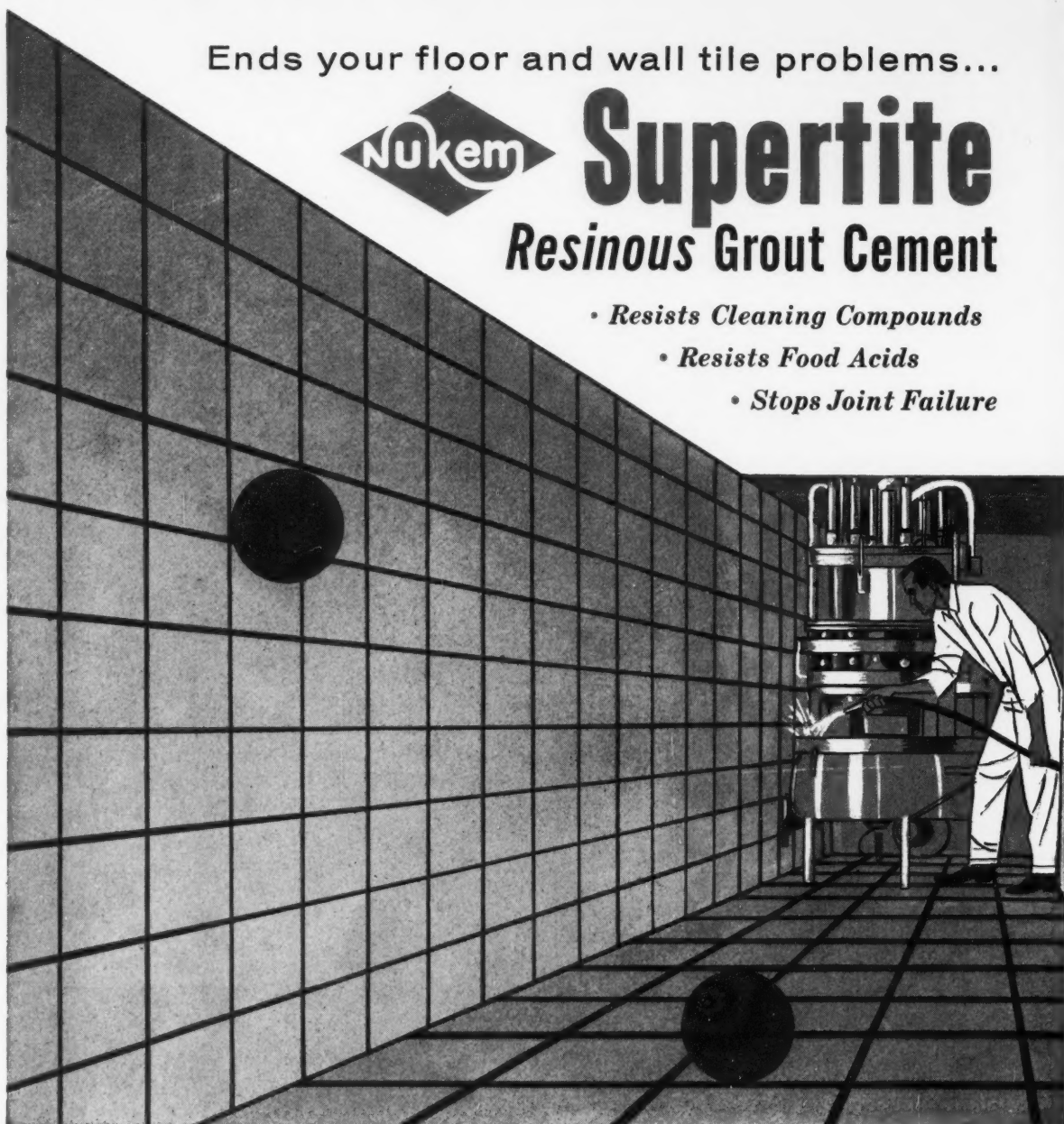
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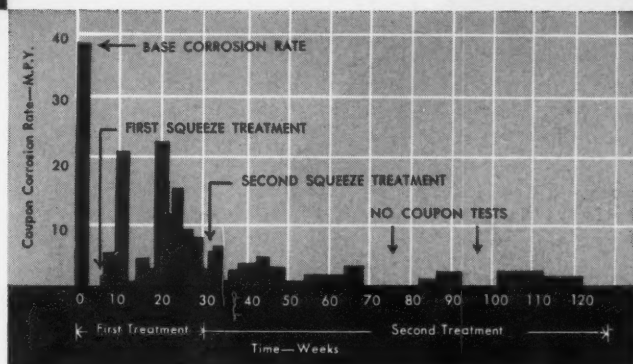
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Corrosion history of a West Texas well,
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Inhibitor squeeze treating began by pumping a mixture of 30 gallons of KONTOL dispersed in 100 bbl. of crude oil into the formation through the casing-tubing annulus. This was followed by a 100 bbl. crude oil overflush. Surface pressure was approximately 1,000 psi during the squeeze. Following this treatment, the well was closed for 20 hours.

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KONTOL results are positive

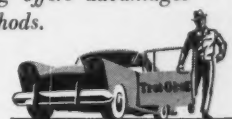
During the first five months following treatment, the corrosion rate varied from a high of 21 m.p.y. to a low of 0.5 m.p.y. For 11 weeks of this period the rate was below 5.0 m.p.y. No equipment failures due to corrosion were reported during the five-month period.

KONTOL protection is long-lasting

Six months after original squeeze treatment the corrosion rate was still satisfactory, but it was decided to resqueeze the well to make certain of uninterrupted protection. During the following 90 weeks, coupon tests showed a corrosion rate that averaged 2.0-3.0 m.p.y.—proof of KONTOL's enduring effectiveness.

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This Month in Corrosion Control . . .

ALLOYS OF FISSIONABLE materials with aluminum and silicon were tested to determine their corrosion resistance in high purity water at 350-360 C. Corrosion of fuel elements accidentally exposed to deionized water in a reactor presents a serious problem because volumetric changes cause the fuel slugs to stick in their channels. Turn to Pages 9 and 12.

TRAINING GAS COMPANY personnel in corrosion prevention involves canny management decisions, if the program is to produce results. Details of one company's program are given in an article beginning on Page 14.

CENTRAL POWER STATION corrosion problems, and some solutions, affecting seizing of turbine control and stop valves, wastage of turbine blades, brass, aluminum and stainless steel heat exchanger tubing, and some design changes are discussed beginning on Page 18.

FLUOROCARBON PISTON RINGS blended with glass fibers, carbon and graphite and flexible hose lined with fluorocarbon resins are discussed and case histories given in an article beginning on Page 26.

SHIP BOTTOM PAINTS can be tested with greater accuracy by a method given in an article beginning on Page 28 than they can with ship trials, authors claim. Advantages claimed for the laboratory tests include better control over conditions and precise timing.

PORTLAND, OREGON will be the scene of Western Region's October 4-6, 1961 annual meeting. A complete summary of the technical program scheduled is given on Page 47.

INTERFERENCE PROBLEMS usually encountered when cathodic protection systems are installed in a congested industrial area are minimized with deep ground beds. How one company solved a particularly knotty problem of this kind is described beginning on Page 75.

PITS IN WATER FLOOD steel injection lines can be anticipated from results of a test described beginning on Page 77. When a flood fluid shows pitting tendencies, most often due to oxygen, a scavenger plus a relatively low inhibitor concentration usually prevents damage. Also described is a low cost, rapid test probe which aids in monitoring the system.

EXTERNAL CORROSION of oilwell casings can be checked and recorded with an eddy current instrument described beginning on Page 81. Logs of downhole runs on 40,000 feet of tubing are summarized. The tool is non-contacting, unaffected by dirt and scale, canting is not significant, 360 degree scanning is obtained, logging at speed up to 3600 feet per hour is possible and it uses a single conductor type logging cable and is independent of fluid in the hole.

CORROSION, July, 1961

SEVERELY COLD WORKED STEEL has a significantly lower desorption rate for hydrogen than material worked less or annealed. This paper, beginning on Page 86, is the third by the same authors on this subject.

CORROSION RATES of carbon steel in boiling solutions of sodium carbonates saturated with carbon dioxide were reduced 96 percent when 0.3 percent hydrogen sulfide was added. For more information on this and other data on stainless steels and Monel in this environment, turn to Page 89.

STRESS-CORROSION CRACKING is reviewed in an article beginning on Page 92. A concise summary of the information currently available on this important corrosion subject.

EFFECT OF TROPICAL environments on stainless steel, part 5 of the series of articles on the effect of tropical environments on metals, begins on Page 97. Eight year tests on six steels in water and atmospheric environments of the Panama Canal Zone are reported. Comparisons are made between performance of the stainless and that of phosphor bronze, naval brass and structural steel.

EFFECT OF IMPURITIES in water on the corrosion rates of aluminum and steel are evaluated beginning on Page 105. The conductance technique was used to determine variations in the corrosion rate.

REDUCING OXIDATION RATE of iron at 750-1025 C with chromium is discussed in an article beginning on Page 109 concerning the rates of oxidation of 0.2 to 10 percent chromium-iron alloys. The growing need for data on performance of materials at high temperatures makes articles of this kind important.

GROUND RODS of mild and galvanized steel, copper clad steel, Ni-Resist and Type 302 stainless will be examined at intervals of one, three and seven years to determine the extent of corrosion suffered when these rods are used to ground neutrals of domestic electrical circuits. A progress report on this program conducted by NACE Task Group T-4A-3 on Methods and Materials for Grounding begins on Page 117.

UTILITY PRACTICES for controlling corrosion of pipe-type electrical transmission cable are summarized in a report by NACE T-4G committee on Protection of Pipe-Type Cables beginning on Page 119. Extensive data on 301 miles of cable installed in the interval 1953-58 are given. An earlier report on this same subject was published in *Corrosion* in 1954.

HULLS OF NAVAL VESSELS have been satisfactorily protected from corrosion for as long as three years with magnesium anodes. Data on this cooperative investigation between the Naval Research Laboratory and the Bureau of Ships will be found in an article beginning on Page 125.



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Growing Interest in Corrosion Control

IT IS EXTREMELY GRATIFYING to note the constantly increasing interest in corrosion control among managers, engineers and scientists all over the world. This interest is demonstrated by the recent formation of associations of persons concerned with the problems in Australia-New Zealand and scheduled organizations of similar groups in other countries. This, added to the accelerated activity of NACE and corrosion control organizations existing elsewhere is strong evidence that the awakening is world-wide.

Not least among the important evidences of interest is the rapidly increasing number of inquiries received by NACE for copies of its technical material and from organizations engaged in studies of the economic potential of various kinds of goods and services suited to corrosion mitigation. Producers of materials, equipment and devices suitable for use in corrosive environments, or who are investigating the advisability of producing such devices—are making inquiries about the economics of corrosion control. Some of these inquiries, considered solely from the implications in the kinds of questions asked, indicate that knowledge of the corrosion control problem is either rudimentary or nonexistent.

In a recent month, requests for copies of literature have been received by NACE from such widely separated places as New York, Portugal, California, Puerto Rico, Belgium, Massachusetts, England, Yugoslavia, Argentina, Canada, Japan, Australia, Pennsylvania, Alabama, New Zealand, France, Norway, Chile, Holland, Israel and Turkey.

Concurrently, there has been an increase in the number of companies assigning to members

of their technical staffs the responsibility of controlling corrosion.

There seems to be solid technical justification for NACE to direct more of its attention to materials other than metals. Deterioration processes of non-metals, while not precisely the same as for metals, often are sufficiently similar to justify adaptation of remedial measures designed originally for metals. Strong evidence is mounting that attacks on non-metallics by corrosives have mechanisms analogous to attacks by corrosives on metals.

No longer is there a clear cut distinction between metals and non-metals as to their properties and reactions. There are plastic nails now which can be hammered through wood. Extraordinarily high strengths are achieved with laminates. New classes of materials based on inorganic structures are contemplated; some are experimentally produced. Performance of materials in extremely high and low temperatures constantly surprises experimenters. Mixtures of metals and non-metals are common; wood impregnated with plastic becomes neither plastic nor wood. Metals are even being mixed (not alloyed) with metals.

These complexities become further involved as improved understanding of properties and reactions leads to changes in previously accepted hypotheses and theories. An example of this is the resurgent interest in ionic states of matter. Another is the growing body of evidence about the influence of electrical and magnetic fields on properties and reactions.

These developments all point unerringly to greater importance for NACE as a tool to reduce corrosion damage. They reiterate the importance of industrial participation in the association's activities, and the value of the association's contribution to the economy.

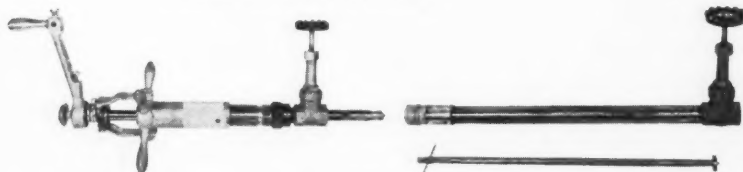
Now! CPS Platinum-Titanium Anodes New Solution to Heater Treater Corrosion

COMPLETE PROTECTION

Titanium has the unique ability to effectively insulate itself and resist corrosion in electrochemical circuits by absorbing oxygen at its surface. When titanium is coated with a layer of platinum, performance is equal to that of a solid platinum anode but at a fraction of the cost.

EASIER INSTALLATION

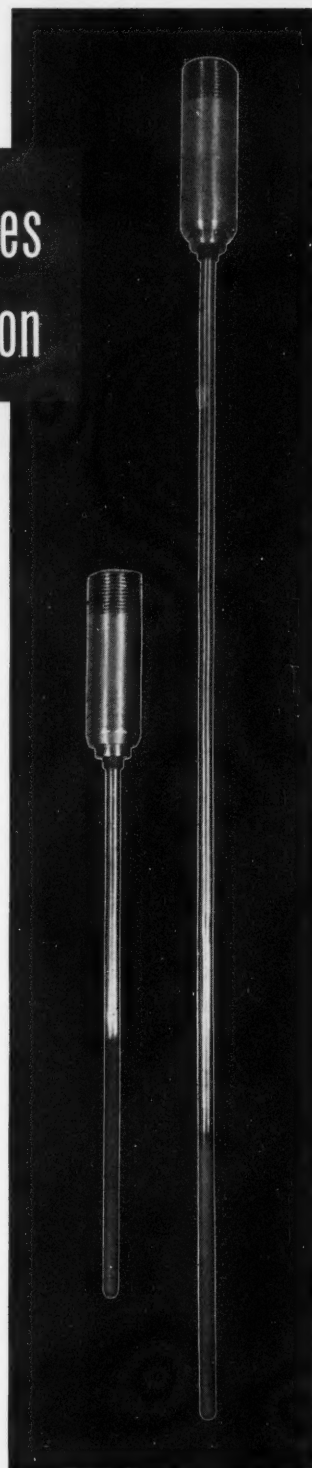
CPS's new small Platinum-Titanium anodes are especially valuable in Heater Treaters, Salt-water Vessels, Heat Exchangers, Water Lines (internal), Pump Manifolds and Process Equipment where space, temperature and corrosive gases make conventional anodes questionable, impractical or difficult to install.



NO DOWN TIME

By using the simple Hot Tapping Tool and Lubricator, hot taps and anode installation can be made without taking existing vessels out of service.

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Introduction

ONE OF the fuel element cores for the Plutonium Recycle Test Reactor at Hanford is a dilute aluminum-plutonium alloy. Little is known about corrosion behavior of this material. The aluminum-uranium and aluminum-plutonium alloy systems present many similarities on the basis of their phase diagrams.^{1,2} While specialized corrosion facilities were being assembled preliminary tests were performed using uranium as a stand-in for plutonium. 1245 aluminum-uranium and 1245 aluminum-uranium-silicon systems were chosen for study because they were most favorable from a nuclear efficiency standpoint. Nickel additions, although they have been found effective in increasing the corrosion resistance of low uranium-aluminum alloys were not made in these preliminary studies.

Experimental Procedure

Preparation of Alloys

Aluminum-uranium master alloys were diluted to the proper concentrations with 1245 aluminum. In the Al-Si alloys, silicon metal was added to bring the silicon content of the alloy to 12 weight-percent. The materials were melted in air and held at a temperature of 750 to

Abstract

Tests in 350 C deionized water of aluminum-uranium alloys were made in an effort to predict behavior in this environment of aluminum-plutonium alloys, corrosion behavior of which is unknown. Aluminum-silicon-uranium alloys also were tested under the same conditions. Samples prepared by casting, of wrought material, and of castings by the cryolite process, were compared. Tests showed no samples significantly distorted jackets when there were minor openings in the jackets. Aluminum alloys with less than 6 weight-percent uranium were unsatisfactory. Aluminum-silicon alloys containing up to 6 weight-percent uranium were satisfactory. Some as-cast aluminum-uranium alloys were preferable to aluminum-silicon. 4.6.5, 6.4.2, 3.5.9

800 C for approximately five minutes. The metal was then cooled to 680-700 C and cast into 6-inch-long rods in a graphite mold. The wrought alloys were worked by hot rolling 0.875-inch diameter rods to 0.6-inch diameter and then machining to 0.5-inch diameter. The test specimens were fabricated by inserting 2-inch-long rod sections into Zircaloy-3 tubing and welding them shut with Zircaloy-2 end caps.

Alloys used in the final test were cast by the cryolite process. Sintered, ball milled UO_2 was charged with aluminum and cryolite (Na_2AlF_6) into a graphite crucible. The crucible was then heated in air to 1050 to 1100 C and held in this temperature range for 20 minutes. The salt layer was then poured off and the crucible allowed to cool to 1000 C. The alloy was then cast into rods in a graphite mold and were water quenched soon after freezing.

Corrosion Tests

Five one-day tests were run on the alloys in 350 C deionized water with a refreshment rate of 1 gph. Aluminum alloys containing 1.6, 4.5 and 6 weight-percent (Continued on Page 10)

* Revision of a paper titled "High Temperature Corrosion of Aluminum-Plutonium and Aluminum-Silicon-Plutonium Alloys" submitted for publication October 13, 1959.



TECHNICAL TOPICS

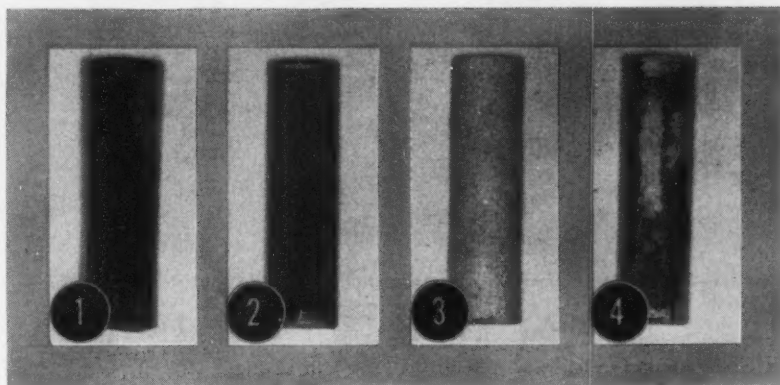


Figure 1—Test One of defected clad pieces. Sample 1: 6 percent uranium in aluminum cast. Sample 2: 1.65 percent U in Al-Si cast. Sample 3: 6 percent U in Al-Si wrought. Sample 4: 6 percent U in Al-Si cast.

High Temperature Aqueous Corrosion of Aluminum-Uranium and Aluminum-Silicon-Uranium Alloys*

H. C. Bowen and R. L. Dillon

General Electric Company
Hanford Laboratories Operation
Richland, Washington

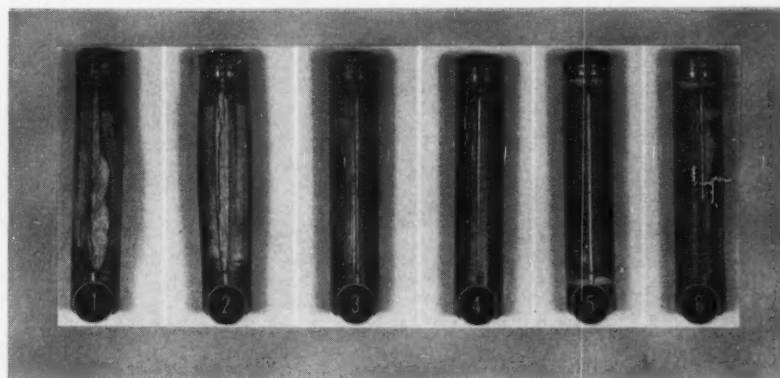


Figure 2—Test Two of defected clad pieces. Sample 1: 4.54 percent U in Al cast. Sample 2: 6 percent U in Al wrought. Sample 3: 6 percent U in Al cast. Sample 4: 1.65 percent U in Al-Si cast. Sample 5: 6 percent U in Al-Si wrought. Sample 6: 6 percent U in Al-Si cast.

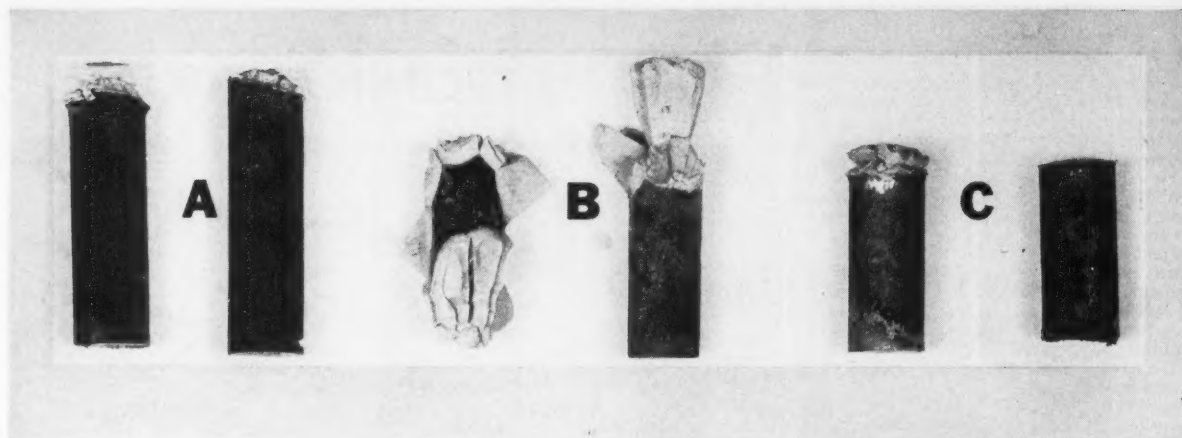


Figure 3—Test Three of higher uranium concentrations. The two samples in each portion (A, B, and C) are 6 percent U on the left and 10 percent U on the right.

High Temperature —

(Continued from Page 9)

percent uranium were tested in the as-cast and wrought condition; both bare and jacketed samples were used. As-cast aluminum alloys containing 1.4, 2.7, 10, 13 and 15 weight-percent uranium were tested bare. Al-12 weight-percent Si alloys containing 1.6 weight-percent uranium were tested as-cast and wrought. The Al-Si alloys were tested both bare and jacketed. Results were interpreted by visual examination to locate samples which, by gross corrosion products,

Results and Discussion

Following is a detailed description of the corrosion tests run and the results obtained. All tests are summarized in Table 1.

Test 1: Defected Clad Pieces

The initial test was of defected clad pieces. A $\frac{1}{16}$ -inch diameter hole was drilled through the cladding of the samples to the alloy core. The samples were autoclaved for 24 hours at 350 C and inspected. Because no dimensional change was noted on any of the samples, they were then autoclaved to a total exposure

of 72 hours. There was still no dimensional change in any of the samples, so they were cut in half longitudinally and the alloy inspected. A light surface oxide was found on all the alloys but there was no evidence of accelerated attack. It is felt that initial corrosion at the defect in the samples formed oxide which "stoppered" the hole in the jacket and prevented more water from coming into contact with the core alloys.

A section from each of the samples was then tested bare for 22 hours in 350 C water. The Al-1.6 weight-percent U and the Al-4.5 weight-percent U alloys, both as-cast and wrought, and the Al-6 weight-percent U wrought alloy were completely destroyed. The Al-6 weight-percent U as-cast alloy showed accelerated attack only on the face which had been worked by cutting. The rest of the sample was covered with a uniform dark grey oxide film. The Al-Si-1.6 weight-percent U as-cast alloy had a uniform oxide film with no evidence of accelerated corrosion. The Al-Si-6 weight-percent U wrought alloy was covered with a heavy white oxide with massive metal underneath. Accelerated corrosion had started on the cut face of the Al-Si-6 weight-percent as-cast alloy. The latter four samples are shown in Figure 1.

Test 2: Defected Clad Pieces

This repeated Test 1 except that defects were made large enough to prevent plugging with oxide. Two slots were cut longitudinally through the cladding to the alloy with a band saw. The pieces were autoclaved for 24 hours in 350 C water. All the Al-U alloys below 6 weight-percent U and the Al-6 U wrought alloys were destroyed and the claddings were either split or badly swollen. The Al-6 weight-percent U as-cast alloy had a very slight swelling at one slot. There was no dimensional change with any of the Al-Si-U alloys. Samples from this test are shown in Figure 2.

Test 3: Higher Uranium Concentrations

Aluminum alloys containing 6 and 10 weight-percent uranium were now tested to determine if the protection gained in the 6 weight-percent uranium as-cast alloys could be extended to higher concentrations. No more Al-Si alloys were tested, as silicon presents a separation problem³ and it would be a great advantage if the same order of corrosion resistance could be attained in Al-U alloys.

A six-inch-long rod of each material was cut into three 2-inch long sections. One end section of each alloy was tested

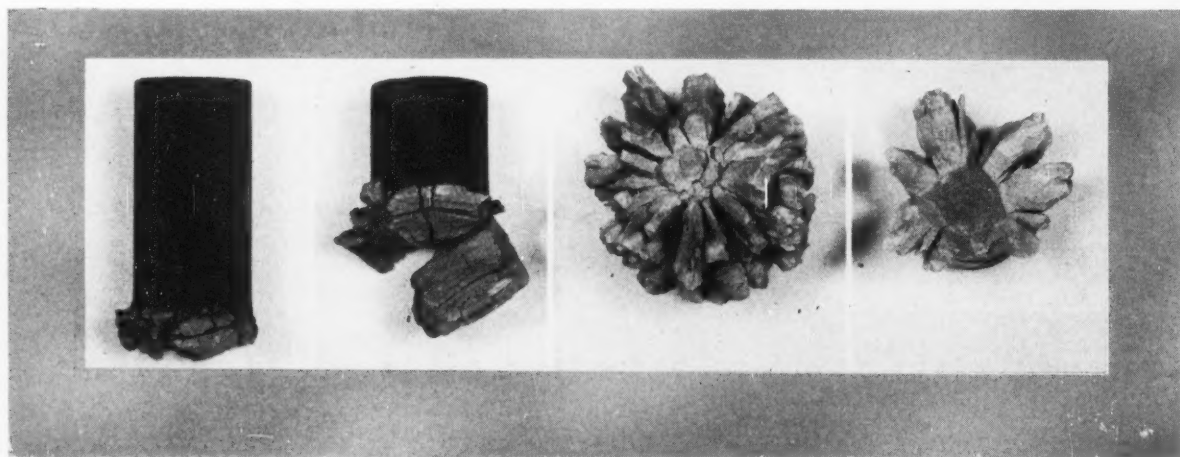


Figure 4—Test Four of cryolite process samples. From left to right the samples are 1.4 percent U, 2.7 percent U, 13 percent U and 15 percent U.

TABLE 1—Summary of Test Results

ALLOY (Weight-Percent)	Condition	Test 1—Bare	Test 2—Defected Cladding
1.6 U in Al.....	Wrought	Destroyed	Can split. Alloy destroyed
1.6 U in Al.....	Cast	Destroyed	Can badly swollen. Alloy destroyed
4.5 U in Al.....	Wrought	Destroyed	Can split. Alloy destroyed
4.5 U in Al.....	Cast	Destroyed	Can badly swollen
6 U in Al.....	Wrought	Destroyed	Can badly swollen
6 U in Al.....	Cast	Accelerated attack on cut face only	Slight swelling at one defect
1.6 U in Al-Si.....	Cast	Surface oxide only	No dimensional change
6 U in Al-Si.....	Wrought	Heavy white surface oxide	
6 U in Al-Si.....	Cast	Accelerated attack on cut face only	
		Test 3—Bare	Test 4—Bare
6 U in Al.....	Cast	Accelerated attack at cut end	Alloy almost completely destroyed
6 U in Al.....	Cast. One cut end etched	Accelerated attack at both ends	
6 U in Al.....	Cast. Homogenized	Accelerated attack at cut end	
10 U in Al.....	Cast	Accelerated attack at cut end	
10 U in Al.....	Cast. One cut end etched	Accelerated attack at unetched end	
10 U in Al.....	Cast. Homogenized	Accelerated attack at cut end. Just starting	
1.4 U in Al.....	Cast. Cryolite process		Alloy almost completely destroyed
2.7 U in Al.....	Cast. Cryolite process		Alloy almost completely destroyed
13 U in Al.....	Cast. Cryolite process		Accelerated attack at cut end
15 U in Al.....	Cast. Cryolite process		Accelerated attack at cut end

without further treatment. The other end sections were heated in an air furnace at 500 C for seven days for homogenization. Fifteen mils of metal was removed from one end of the center sections in a H_3PO_4 - H_2SO_4 - HNO_3 chemical polishing bath to remove the cold work. The samples were tested for 24 hours in 350 C water.

Samples tested without special treatment verified the results obtained in Test 1. The pieces corroded rapidly at the cut end, but showed no accelerated attack on the remainder. The 6 weight-percent samples showed more severe attack, indicating greater sensitivity to cold work (Figure 3A). Sections which had 15 mils of metal removed from one end showed also that the 6 weight-percent U alloy is more sensitive to cold work. Though the samples received the same amount of cold work during cutting and the same amount of metal was removed from both alloys, the 6 weight-percent U alloy corroded rapidly at both ends while the 10 weight-percent U alloy corroded rapidly at the untreated end only (Figure 3B). Homogenization definitely improved resistance of the samples. Although the 6 weight-percent U sample showed accelerated attack at the cut end it was not as severe as in the other 6 weight-percent U samples. The 10 weight-percent U sample showed very little attack anywhere on the sample (Figure 3C).

Test 4: Cryolite Process Samples

Samples used in this test were rod ends from alloys cast by the cryolite process for comparison with normally cast material. Higher uranium concentrations also were available in this material. The test was again for 24 hours in 350 C water. Results of the other tests were confirmed, i.e., the difference in method of preparation was not a significant factor in corrosion behavior. Alloys containing 1.4 and 2.7 weight-percent uranium were almost completely destroyed. Alloys containing 13 and 15

weight-percent uranium corroded rapidly only at the cut, cold-worked ends (Figure 4).

Conclusions

From these tests, the following conclusions can be drawn:

1. None of the clad aluminum-uranium or aluminum-silicon-uranium alloys corrosion tested with a $\frac{1}{16}$ -inch diameter defect in cladding distorted the jacket enough to reduce water flow in a process-tube.
2. Aluminum alloys containing less than 6 weight-percent uranium are not satisfactory high temperature fuel element cores from the standpoint of swelling in case of an extensive jacket failure.
3. Al-Si alloys containing up to 6 weight-percent uranium are satisfactory. Higher uranium contents probably also are satisfactory but were not tested.
4. As-cast aluminum alloys containing from 6 to 15 weight-percent uranium are comparable to the Al-Si alloy and are preferable to Al-Si because of the separations problem presented by silicon.
5. In no case was the attack as severe on the aluminum-uranium alloys as it would be on metallic uranium.

Acknowledgments

Alloys used in all tests but one were cast by C. H. Bloomster of the Plutonium Metallurgy Operation. Alloys for the final test were cast by W. L. Lyon of the Chemical Research Operation.

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High Temperature Aqueous Corrosion of Aluminum-Plutonium and Aluminum-Silicon-Plutonium Alloys*

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Hanford Laboratories Operation
Richland, Washington

Abstract

Aluminum-plutonium and aluminum-silicon-plutonium alloys were tested in 350 and 360 C deionized water in a static autoclave. Alloys were air-melted and cast. Tests showed low percentage aluminum-plutonium alloys had little resistance to corrosion but that resistance increased with increased plutonium. Working the material drastically decreases resistance. Silicon additions make some alloys resistant to attack for at least 24 hours and also less sensitive to working.

6.4.2, 4.6.5, 3.7.2, 3.5.9

Introduction

ONE of the proposed fuel cores for the Plutonium Recycle Test Reactor at Hanford is an aluminum-plutonium alloy. The corrosion behavior of this class of alloys in high temperature water has not been determined, though the behavior of the similar aluminum-uranium system has been described.¹

Two alloy systems, 1245 aluminum-plutonium and 1245 aluminum-silicon-plutonium were chosen for investigation because they are most favorable from a nuclear efficiency standpoint. Nickel additions, though effective in increasing the corrosion resistance of the one to two percent uranium-aluminum alloys were not made in these preliminary studies.

Experimental Procedure

Alloys all were air-melted in clay-graphite crucibles in a resistance wound furnace. Raw materials used were 1245 Al, commercial eutectic Al-Si alloy and metallic Pu. Melts were held at temperature and stirred manually until complete solution was obtained. Casting temperatures generally were 50 - 75 C above liquidus. No flux was used in fabrication. Alloys were cast into 1/2 or 5/8-inch diameter warm graphite molds with a wall thickness of 1/2-inch. Alloys were radiographed to detect porosity and segregation and insure that samples were of the required quality. See Table 1.

Samples to be tested were prepared by one of the following methods:

1. A 2-inch long end section was cut from a 6-inch long cast rod. The cut end was etched in a 78 percent commercial H_2PO_4 , 11 percent commercial HNO_3 , 11 percent commercial H_2SO_4 , and 0.8 g/l $FeSO_4 \cdot 7H_2O$ chemical polishing bath. This was done to remove the cold work due to cutting. This left an as-cast and an etched cut end on the sample.
2. A 2-inch long section was cut from the center of a 6-inch long cast rod. One end was treated in a polishing bath as above. This left a cut end and an etched cut end on the sample.
3. Two-inch long sections were cut from 1/2-inch diameter extruded rod and tested without further treatment.

A simple static autoclave installed in a hood to prevent spread of contamination

tion from the metal and corrosion products was used.

No quantitative measurements were attempted and all results were interpreted by visual observation.

Results and Discussion

Tables 2 and 3 give results of samples tested in 350 and 360 C static deionized water respectively. They bear out results obtained in the tests of the aluminum-uranium alloys in all cases. The two alloy systems seem comparable under the conditions of test. Temperature dependence of the attack indicated by comparison of results at the two temperatures is greater than one would expect from a 10 degree differential. However, this may be an alloy effect or, as the 360 C tests were the first run in new equipment, it may be an operating variable.

Conclusions

The following conclusions may be drawn from these tests:

1. Low percentage (6 percent) aluminum-plutonium as-cast alloys have little resistance to rapid high temperature attack but resistance increases with increasing plutonium content up to at least 13 percent Pu.
2. Any working of the material drastically decreases its resistance to attack as compared to as-cast material of the same Pu content.
3. Addition of 12 percent silicon to aluminum-plutonium alloy makes the alloys up to at least 15 percent Pu resistant to attack for at least 24 hours and makes them less sensitive to working.

Acknowledgment

Alloys used in these tests were cast by C. H. Bloomster of the Plutonium Metallurgy Operation.

References

1. Intergranular Corrosion of Aluminum-Uranium and Aluminum-Silicon-Uranium Alloys, H. C. Bowen and R. L. Dillon, HW-55352, 3-14-58.

TABLE 1—Description of Alloy Preparation

Alloy*	Max. Melt °C	Soak Time (hrs)	Casting °C
Al-Si-Pu			
2 Pu.....	925	2	700
5 Pu.....	900	2	725
10 Pu.....	900	1 1/2	775
15 Pu.....	950	4	850
Al-Pu			
To 15 Pu..	900	4	730

* Weight percent.

TABLE 2—Rapid High Temperature Attack by Al-Pu Alloys in 350 C Water

Wt. Percent Pu	Time (hr)	Sample Treatment (see text)	Results
13	24	1	1/4" D spot attacked on etched cut end.
13	24	2	1/4" D spot attacked on both ends.
10	24	1	Not attacked
10	24	2	Both ends and one side attacked
6	24	1	Not attacked
6	24	2	Badly attacked on cut end and one side
1.8	24	1	Destroyed
1.8	24	2	
10	3	3	Approximately 1/2 of diameter lost
10	3	3	
1.9	3	3	Destroyed
1.9	3	3	

TABLE 3—Rapid High Temperature Attack of Al-Pu and Al-Si-Pu Alloys in 360 C Water

Wt. Percent Pu	Time (hr)	Sample Treatment (see text)	Results
13	24	1	Etched cut end attacked
13	24	2	Both ends attacked and 1/4 of sample destroyed
10	24	1	Etched cut end attacked, 2 small spots on side attacked
10	24	2	Cut end and one side of sample attacked and 1/4 of sample destroyed
6	12	1	Etched cut end attacked and 1/2 of sample destroyed
6	12	2	
1.8	12	1	Destroyed
1.8	12	2	
15*	24	1	Not attacked
15*	24	2	
10*	24	1	
10*	24	2	
5*	24	1	
5*	24	2	
1.8*	24	1	
1.8*	24	2	

* In Al-12% Si

★ Submitted for publication October 13, 1959.



BETTER COATINGS THROUGH RESEARCH

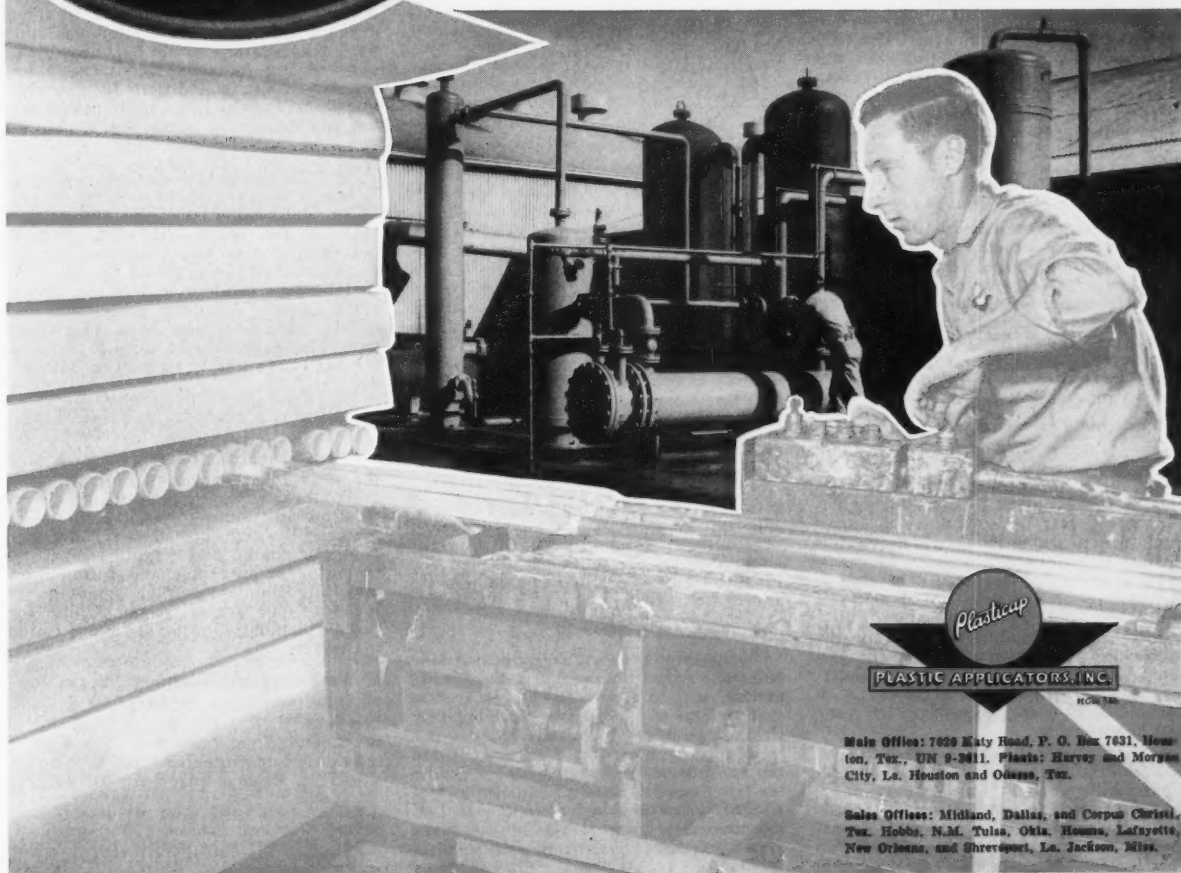
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CORROSION CONTROL efforts of a company can be made more effective through a training program based on the company's particular training needs if the program is carefully planned, effectively conducted and properly evaluated.

This article describes the experience of Oklahoma Natural Gas Company in the use of a training program to improve its personnel's cathodic protection installation and maintenance work. The program was begun after a test given to company personnel showed that they did not fully understand the importance of proper installation in cathodic protection work.

Planning a Training Program

The essential first step in launching a training program, whether in corrosion control or any other field of work, is to obtain a realistic survey of training needs and to analyze them in terms of scope and type of training required. Only after specific needs are known can an effective and economical training program be organized. Before an individual worker can be trained to improve his work, his supervisor must determine what the worker's performance should be in comparison with what it is.

Unless proper planning is an integral part of a training program, inadequate training may result. Borrowing training programs from another company, even though it is a successful program, may not satisfy the needs of a specific company.

The story is told of a meeting of men interested in a particular training program in which one man was asked, "How did you get your program started?" He replied, "We borrowed it from the telephone company." The telephone company representative who was present admitted, "We borrowed it from a gas company." The gas company representative astonished the group by saying, "The fact is, we discarded that program about two years ago because it did not produce results."

Principles Involved in Training

A training program should be geared to meet current and projected manpower needs. Too much training is just as wasteful as too little. The program should be tailored to individual requirements and potential abilities because all employees cannot be trained to a common level of performance. Each person possesses different attitudes, interests and learning ability.

Determining Training Needs

The process of determining training needs involve two basic steps: (1) recognition of the problem area and (2) analysis of the problem to determine what part of it can be solved by training. Such needs are obvious when the following situations are found: excessive rework, expanded operations, new operations or procedures, conversion of equipment and high accident rate.

Also, some conditions such as the following may involve hidden or obscure needs for training: general lowering of performance standards, high maintenance costs, excessive overtime and bottlenecks.

Several methods can be used to determine training needs such as conferences with various levels of management, analysis of performance records and reports on maintenance and operating costs, questionnaires based on specific job requirements and opinion

Cecil Gibson

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Tulsa, Oklahoma

Abstract

Describes one gas company's experience in planning and setting up a training course for its personnel in cathodic protection installation. Explains importance of planning a training course to achieve desired results. Outlines principles of training, methods of instruction used, subject matter of course and methods of evaluating course. Presents a 51-question test on cathodic protection and corrosion control in general which was given to company personnel to determine need for training and also used after the training course to evaluate how much knowledge had been acquired by participants.

1.7.1, 5.2.1

Gas Company Uses Cathodic

surveys conducted by outside consultants.

Top Management Support Necessary

The support of top management is necessary in any successful training program. If a company officer is asked, he generally will reply that employee training and development is a must and that he will support such activities. This favorable attitude of top management when communicated throughout the company helps stimulate participation from supervisors and employees to achieve the training objectives.

Importance of Evaluation

Evaluation of any training program is necessary to determine if desired objectives have been fulfilled. Most training programs can be evaluated in several ways. The following three points should be included: (1) study of conditions before the training program is begun, (2) determining what conditions are desired after the program is completed and (3) comparing conditions before with conditions after training program is completed.

Cathodic Protection Training Program

The first step taken at Oklahoma Natural Gas in planning its training program was a meeting of operating superintendents and key supervisors to determine the training needs of the company. Among the subjects selected for the program were corrosion in general and cathodic protection.

Quiz Given

To pinpoint specific training needs and to verify the need for a training course, a quiz on cathodic protection and general corrosion was developed by the company's general director of cathodic protection and five district cathodic protection inspectors and the training coordinator. The quiz (see Figure 1) contained 51 questions including multiple choice, true-false and essay type questions based on job requirements of construction foremen, line

maintenance foremen and servicemen.

When the quiz was given, its purpose was explained: to pinpoint those areas of corrosion control and cathodic protection in which personnel needed a better understanding so that course material could be developed for the training program. It was explained that no grade would be reported on the quiz, but each question would be individually checked and analyzed. Personnel taking the test were asked not to guess at questions to which they did not know the answer.

Results of Quiz

Analysis of the test showed 20 percent incorrect answers on the multiple choice, 22 percent on the true-false and 45 percent on the essay questions. Highest percentage of incorrect answers on the essay questions established the fact that there was considerable lack of understanding why cathodic protection materials were installed in a prescribed way. This established a need to improve the foreman's ability to instruct his personnel.

Based on 400 people who took the test, there was an average of 25 percent of the questions missed.

Further analysis of the tests showed the need for clarification of instruction in bonding wires to pipe, installing insulation in domestic and commercial meters, making proper field reports, handling coated and wrapped pipe and fittings, installing mechanical couplings, installing anodes at pipe maintenance location and fixing responsibility for proper installation of cathodic protection materials.

Preparing the Training Program

After the tests were analyzed, a meeting was held with the general office management personnel and each district operating superintendent and key supervisors to discuss plans for developing

★ Revision of a paper titled "How to Improve Your Corrosion Prevention Through Training," presented at a meeting of the Central Oklahoma Section, National Association of Corrosion Engineers, December 12, 1960.

to improve installation operations

the scheduled date of the course.

It has been found that, by giving the people their quizzes a week or ten days before the course, they became familiar with the specific areas missed on the tests. This also stimulated their need for the course; thus they were especially alert to the instructor's remarks in those areas in which the individuals needed a better understanding.

The course outline provided the participants sufficient time to think through the subject matter before attending the course. Thus, they were able to help make the course more interesting and

Evaluation of the Training Course

Immediately after the last session in the course, the quiz given before the program was given again so that an immediate evaluation could be made of what the participants learned by comparing their scores on the two tests. Also, the second test gave an indication of areas where additional training may be needed.

Comparison of the pre-instruction and post-instruction tests showed a 90 percent increase in the number of correct answers on the post-instruction test.

c Protection Training Program*

the course material, selection of instructors, length of time required to complete the course, location of individual sessions and evaluation procedures.

The company's general cathodic protection director and the district cathodic protection inspectors were used as instructors. Their selection was based on familiarity of the subject matter, previous experience as instructors and their experience in dealing with people, procedures and training problems. These instructors met with the company's training coordinator to review the instructor's outline of the course, improve instruction techniques, develop visual aids and establish the area which each instructor would cover. After each instructor gave a 30-minute presentation of the area assigned to him, a critique was held to point out good points and to give suggestions where improvements could be made to stimulate more interest and participation. Tape recordings of these presentations also helped the instructors improve their teaching.

Preparation guides were given to the operating superintendents to help them stimulate interest among personnel in the various departments. The guide presented information and data on the cost of installing and maintaining cathodic protection on the company's underground structures. Costs were given and explained on the waste and inefficiency resulting from improper handling, coating and installation of cathodic protection materials. This wasted money was related in terms of how it affected both the company and the employees.

The information in the guide was used by the operating superintendents in writing a memorandum to the personnel selected. They were encouraged to attend the course with an open mind and a desire to learn and to think continuously of how they were going to apply their classroom learning experience to application on the job. This memorandum, the quiz and the course outline were forwarded to each person about a week or ten days before

educational through their participation and eliminated questions and discussion on topics out of chronological order.

Training Aids Used

Training aids used in the course included 35mm slides, flip charts, white pads and a soil box demonstration. The slides covered such topics as machine and hand coating process, meter installed in a bind, foreign lines layed in contact with company lines, improper handling of coated materials such as pipe, valves and fittings and procedure for anode installation.

Flip charts were used to show the fundamental theory of cathodic protection and the mechanism of corrosion on underground structures. Instructors agreed that the white pad can be used advantageously to focus attention on the topic being discussed, to provide a permanent record that can be used in reviewing the course and to encourage participation by listing the answers from the participants.

The soil box demonstration was made with transparent meter and overhead projector, similar to demonstrations given at several NACE meetings and short courses throughout the country.

Good training aids are not a substitute for an instructor, but should be used to improve the presentation.

A glossary of technical terms used in the training course was given to participants for a better understanding of the subject matter.

Pilot Session Held

Before the course was given in the company's five districts, a pilot session was held to determine if the course needed changes to make it more effective and to determine the number of hours required to complete the course. This pilot session also gave the instructors another opportunity to evaluate and improve their instruction techniques. A minimum of eight hours was decided as the time required to cover the course material.

After the quiz, each participant was given a comment sheet to be submitted to the training coordinator within three or four days. This sheet was designed to measure the participant's reaction to the course and to provide a basis for improving the course.

Follow-Up on the Course

All too often, a training program can be considered a success, but the learning acquired in the program is not used on the job by personnel. An informal discussion between the supervisor and his personnel is recommended to provide an incentive for the participants in a training course to use their learning experience on the job.

About four months after the training course is completed, each supervisor and district cathodic protection inspector makes an evaluation of the training program in terms of improvement in handling, coating and installing of cathodic protection materials.

One result of the analysis of the quizzes and comments from construction foremen, serviceline foremen and servicemen is preparation of a new foreman's manual. Purpose of the new manual is to clarify existing policies and procedures and to standardize others. The manual also will be used in training personnel.

see page 16
for
training
program
test

CATHODIC PROTECTION TEST QUESTIONS

True or False Questions (If any part of statement is false, mark it false.)

1. In the event that galvanized and black pipe are coupled and installed in the ground, the black pipe will corrode first.

2. All Style 38 Dresser couplings should be bonded.

3. The crew that installs pipe should also install the anodes.

4. When two pipes are connected with an insulating coupling, it is essential that they should be in almost perfect alignment.

5. It is not necessary to coat non-insulating mechanical couplings.

6. All insulating mechanical couplings should be coated.

7. According to company policy, a construction job is not considered complete until cathodic protection is established.

8. When cutting a line that has a rectifier, all that is necessary for safety is to turn the rectifier off.

9. It is impossible to cathodically protect a pipe inside a "shorted" casing.

10. Meter insulation should be placed on the inlet side of meter settings.

11. A pipeline is considered "shorted out" when the coating has been damaged.

12. When company lines are laid on a foreign line (such as water service) or when a foreign line is laid on a company line, the pipe coating is not sufficient to prevent the eventual electrical contact.

Essay Type Questions

1. If bare and coated pipe is coupled with an insulating coupling, on which should the boot be installed? Why?

2. What type of soil is best suited for anode installations? Why?

3. Why do most leaks appear on the bottom half of the pipe?

4. What size anode should ordinarily be installed at a pit hole leak repair? On a main? On service renewal?

5. Why is it necessary to insulate bare pipe from coated pipe?

6. What safety measure should be observed to prevent arcing when cutting a pipeline?

7. How many casing insulators should be used in a 60-foot casing?

8. What size casing should be used with the following: 1 1/4-inch carrier pipe, 2-inch carrier pipe, 4-inch carrier pipe, and 8-inch carrier pipe?

9. What single item is most important to a good coating job?

10. What information is required to properly fill out the "Cathodic Protection Copy" of a job order?

11. What testing procedure should be followed after installing a Style 39 insulated Dresser coupling?

12. What steps should be taken before lowering in-yard coated pipe in which a cold bend has been made?

13. Describe the procedure necessary to get a good coating job using cold applied tape and primer.

14. When installing insulating meter swivels, is it necessary to use a rubber gasket? Why?

15. When changing meters, should you change out the old style insulating meter swivels? Why?

16. What record should you make of anodes installed on services?

17. Why should insulating connections be painted black?

18. Please list specific things about cathodic protection for which you would like to have additional information.

Multiple Choice Questions

1. Corrosion is most likely to be found in (a) sandy soil, (b) loam soil, (c) clay soil.

2. A galvanic corrosion cell is caused by (a) insulating flanges, (b) coupling of unlike metals, (c) holidays in coating.

3. Wires should be installed on pipes by (a) brazing, (b) thermite welding process, (c) band clamps.

4. Damage to pipe resulting from wrench marks, shovel marks, etc., causes corrosion due to (a) soil changes, (b) stress points on pipe surface, (c) increased oxygen content.

5. In the event that an insulating coupling should be "shorted" on a new installation due to improper installation, who should re-install it? (a) cathodic protection inspector, (b) district engineer, (c) original installer.

6. Meters which are installed in a bind may destroy the effect of cathodic protection because (a) thread leaks may develop at the return bends, (b) meter insulation may be damaged, causing it to short out, (c) unnecessary strain is placed on the meter casting.

7. Voltages that could cause electrical shock to a person at insulating meter settings originate at (a) anode installation, (b) rectifiers, (c) alternating current systems.

8. The purpose of an insulating coupling is to (a) insure electrical continuity, (b) isolate one section of pipe from another, (c) create an expansion joint in the line.

9. The pipeline easiest to protect with cathodic protection is (a) a well-coated and insulated line, (b) a bare line, (c) a well coated line not insulated.

10. The best method to haul coated pipe to the job is (a) boomed down to the headache rack, (b) fastened in a pipe vise, (c) padding between layers and under boomer chains.

11. Gate boxes and underground meter boxes should not be in metallic contact with company lines because: (a) their weight may break the pipe, (b) they may damage the pipe coating, (c) they may short-out the insulation.

12. All underground gate valves should be insulated in order to (a) eliminate use of anodes, (b) keep them from freezing in winter, (c) electrically isolate sections of pipe.

13. When a test wire is needed for testing pipe-to-soil voltage potential, it should be placed (a) as near to anode connections as possible, (b) at opposite end of line from anode connection, (c) near a meter riser which is connected to line.

14. When an insulating coupling is installed, the pipe ends being joined should (a) both be threaded to give it strength, (b) be threaded on end where plastic sleeve is installed to hold sleeve, (c) be plain on both ends.

15. The proper way to check an insulating meter swivel for electrical continuity with a test light is (a) touch two contact probes to meter risers and see if light shines, (b) use above procedure plus compass on top of meter, (c) place compass under the meter.

16. When an anode is installed on new coated, wrapped and insulated pipe, water is used to (a) make the anode easier to slide in augured hole, (b) saturate the magnesium to give it longer life, (c) wet the backfill to get instant action from anode.

17. The proper way to install a magnesium anode in the soil is to (a) lay the anode down in bottom of bell hole, (b) auger a hole approximately the same size as anode, (c) stand the anode against the side of the bell hole.

18. The best place to install an anode on a pipe renewal is (a) at the leaking area, (b) in the bell hole at the tie-in, (c) in the middle of the pipe installed.

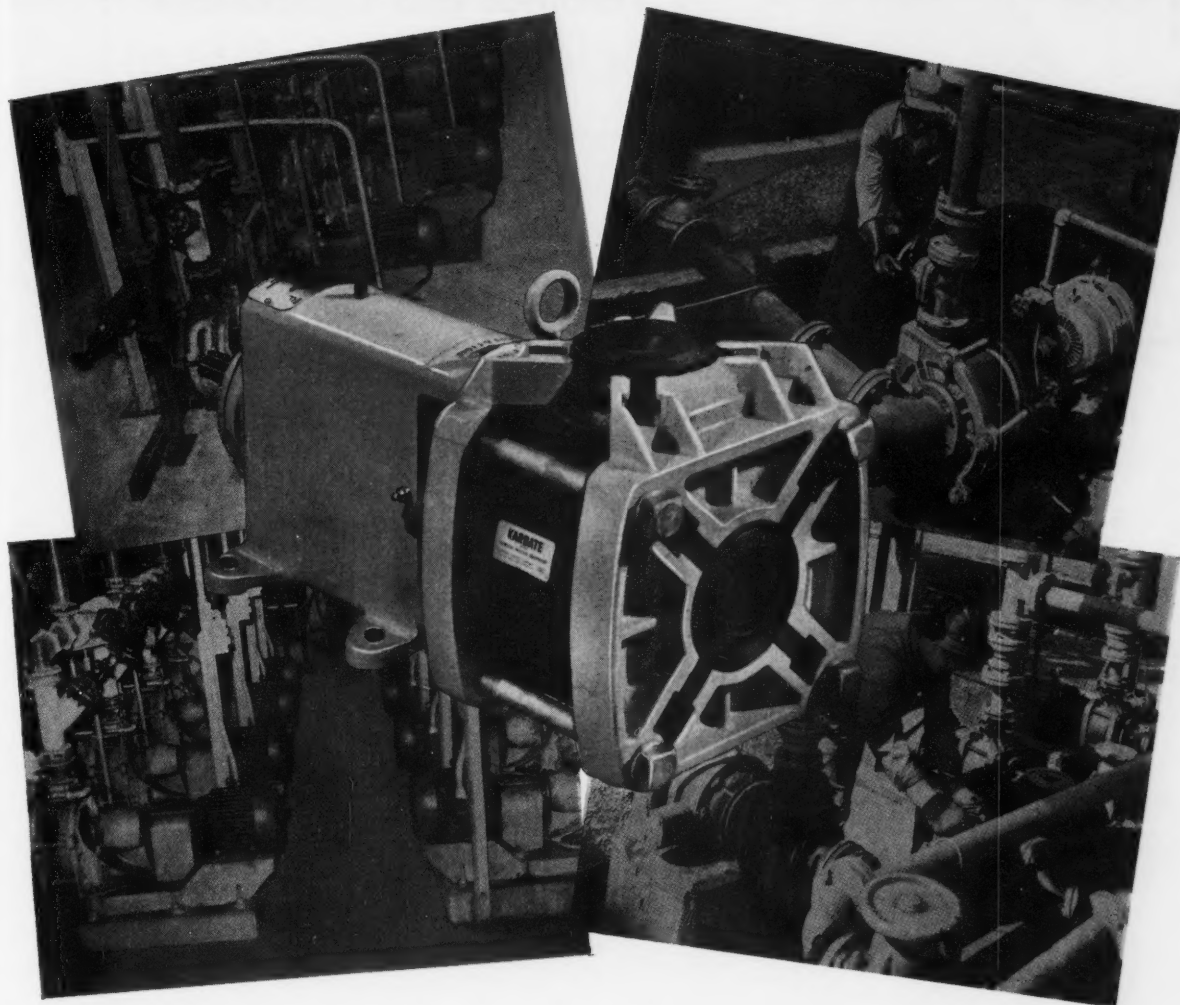
19. In distribution, all anode measurements should be from: (a) a residence property line, (b) the street curb line, (c) the center line of the city street.

20. When installing an insulating joint above ground, you should (a) paint it black, (b) coat it with aluminum paint, (c) never paint it.

21. When a 1 1/4-inch insulating coupling is installed, the pipe gap should be about (a) 1/8-inch, (b) one inch, (c) 4 inches.

FIGURE 1—Cathodic protection test used to plan and to evaluate a company's training program.

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Corrosion Problems of Central Power Station Turbines and Condensers*

A. Bayard Sisson

Commonwealth Edison Company
Chicago, Illinois

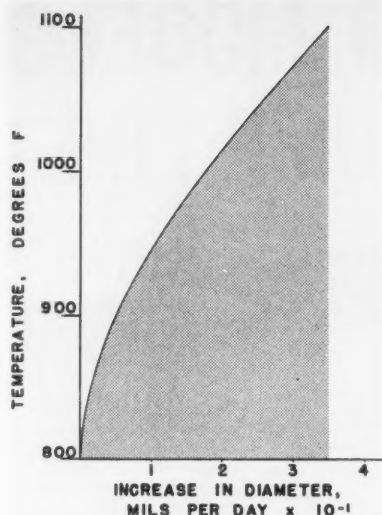


Figure 1—Oxidation of nitrated Nitralloy in steam temperature dependent function.

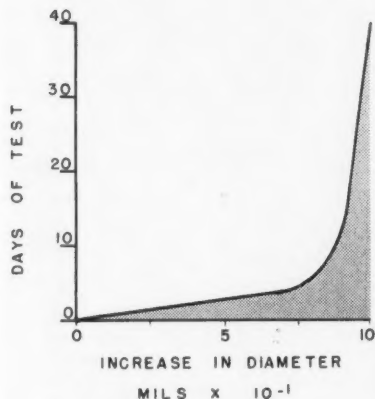


Figure 2—Oxidation of nitrated Nitralloy in steam at 1000 F time dependent function.

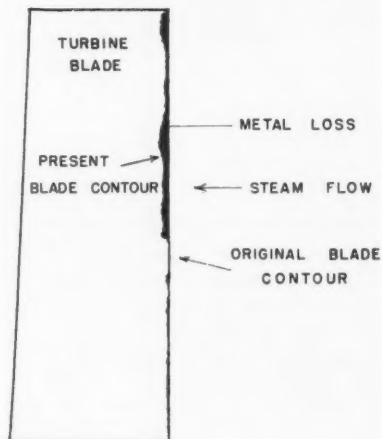


Figure 3—Shadow graph of a turbine blade from one of last two or three rows of blades at exhaust end of a large condensing turbine.

Abstract

Experience with Commonwealth Edison generating plants in the Chicago area are discussed. Dangerous seizing of turbine control and stop valves at temperatures over 900 F was solved in part when a research program disclosed nitrated surfaces were corroding to cause the failures. Research on the problem continues. Turbine blade failure, originally attributed to corrosion fatigue, was discovered to be the result of inadequate fatigue limit of the blade material and not sodium hydroxide carry-over.

Influence of noncondensable gases which cannot be completely removed from boiler water is discussed. Wastage of turbine blades was attributed to corrosion-erosion where steam and water both are present. Gases responsible for the accelerated attack were oxygen, carbon dioxide and sulfur dioxide at low pH values.

Method of using morpholine to control alkalinity is discussed. Experience

with admiralty brass exchanger tubes is reviewed, including descriptions of difficulty in "air removal" sections where noncondensable gases were found to be troublemakers. Ammonia and sulfur dioxide tend to concentrate in the cooled water in these zones sometimes to concentrations as high as several hundred parts per million at tube surfaces. Resulting pH values caused high removal of copper. Solution of this problem is elusive, although removal of some of the tubes in the affected region is being tried. Another solution is replacement of admiralty tubes in this section with Type 304 or 316 stainless steel. Alarming losses from tube metal by erosion are reported.

Aluminum tubes, installed in one condenser, had to be removed in three years. In another test 6061 aluminum tubes lasted 5500 hours while 3003 lasted only 3500. In comparison with brass tubes, whose service life is expected to be about 30 years, the aluminum did not give adequate performance. 8.2.2

Introduction

THE TRUE DEFINITION of corrosion probably is not broad enough to encompass all conditions described in this discussion. However, most of the problems encountered do have their origin in a true corrosion phenomenon and therefore relate directly to corrosion engineering.

Condensers are physically connected and what is much more important, are continuously confining the same media, steam and steam-water mixtures.

Among the metals involved in the turbine-condenser structures are cast irons, carbon steel and stainless steels; hard, wear-resisting alloys, copper alloys and aluminum. Turbines concerned vary in size from 5000 KW to 325,000 KW and the steam conditions vary from 300 psi at 600 F to 2000 psi at 1050 F. Condenser sizes vary accordingly and are all on fresh water sources. The discussion is confined to the shell side of exchangers because this side has the same environment as the turbine.

Chemical treatment or the lack of it has much to do with conditions governing corrosiveness of steam and steam water mixtures in the steam turbine and its associated condenser. The Commonwealth Edison Company has used a type of chemical treatment which has given

excellent protection of the entire system, ie., condensate, feedwater, boiler, turbine, condenser and feedwater heaters. Quantities of chemicals maintained in the different portions of the cycle vary somewhat as pressures and temperatures change but the base treatment remains the same. Boiler water is treated to maintain moderate residuals of phosphate and sodium sulfate, minor hydrate alkalinity and a smaller excess of sodium sulfite, the whole producing a pH of from 10.5 to 11.0. Sodium sulfite is fed continuously in proportion to the quantity of feedwater and to the dissolved oxygen content of this water. The other materials may or may not be so fed. An amine, morpholine, also is continuously fed to maintain an excess in the steam and feedwater portion of the cycle, this excess being sufficient to give a pH of 8.8 to 9.2.

Make-up water is, in most cases, supplied by demineralizers and is a very high quality water containing only fractions of parts per million of dissolved solids. Evaporators still are used in a few of the older stations where steam pressures and temperatures are in the low or intermediate range.

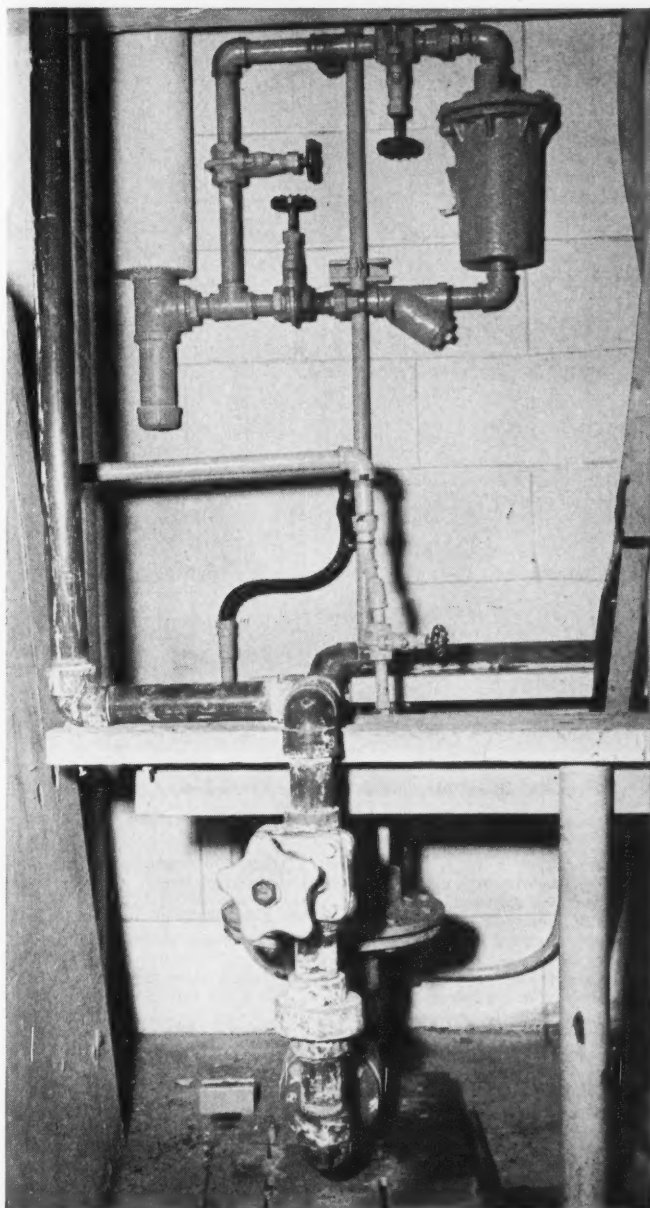
Problem of Seizing Control Valves

A serious condition which has plagued operation of turbines involves turbine control and turbine stop valves, which after varying periods of service become immobile and will not function as designed. Under some circumstances a disastrous turbine runaway could occur. (Continued on Page 20)

* Revision of a paper titled "Corrosion of Steam Turbines and Condensers in Central Station Power Plants," presented at a meeting of the North Central Region, National Association of Corrosion Engineers, Milwaukee, Wis., October 19-20, 1960.

METAL REFINING: New Role For Penton*

Solid Penton pipe and fittings prove easy to install and maintain, and low in cost



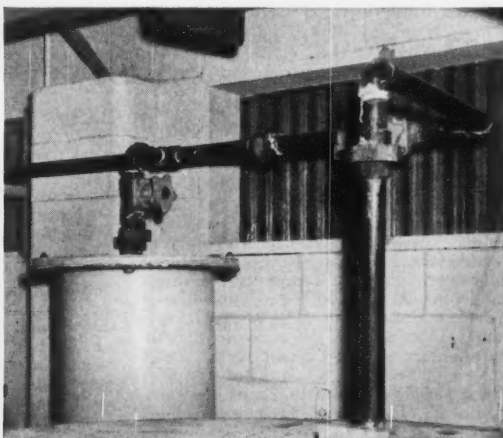
The Wah Chang Corporation has found an important new use for Penton at its Albany, Oregon, plant where columbite and tantalite ores are refined to obtain columbium and tantalum.

More than 100 feet of 1½ inch Schedule 80 pipe, threaded tees, 90° elbows, unions, and nipples—all made of solid Penton—are serving in transfer lines in the Wah Chang process, where columbium and tantalum are separated by liquid-liquid extraction and the oxide products are reduced to metal by carbon reduction.

In selecting the process equipment, Kenneth W. Bird, Process Engineer, says: "The extreme ease of installation and comparative cost of Penton pipe and fittings versus other materials which could handle this rough piping job, make Penton by far the most advantageous material we could employ. Our product must be of the highest purity, and Penton exhibits the greatest degree of inertness to the materials being handled of any competitive product on the market."

Write for complete facts about Penton and a list of fabricators and suppliers of Penton processing equipment.

Pipe and fittings used at Wah Chang were supplied by Tube Turns Plastics, Inc., Louisville, Ky., through its West Coast distributors, Esco Corporation.



At left: Discharge side of diaphragm pump in which hot (210°F.) 15% HF solution and K₂TaF₇ (Potassium fluo tantalate) is transferred to crystallization tanks through Penton system of 1½" Schedule 80 pipe and threaded fittings. Above: Transfer line into crystallization tanks is solid Penton pipe.



*Penton is the Hercules Powder Company registered trademark for chlorinated polyether.

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Central Power—

(Continued from Page 18)

Valve stems and associated guide bushings grow in size until there is no clearance between them, so they no longer operate. This condition became noticeable as operating temperatures increased. At 900 F it was only a minor nuisance, at 950 F it became an annoyance and at 1000 F and 1050 F it developed into a serious condition.

Turbine manufacturers "consoled" the operators with the explanation that the condition resulted from soluble or insoluble iron oxide carryover with high temperature steam with resulting deposition on valve stems and bushings. The conclusion was that there was no real cure and that periodical maintenance was required to re-establish the required clearances between the various components.

Research Program Seeks Solution

This author, after observing many valves and bushings, decided that these conclusions were assailable and directed his ideas to a research program to determine the cause of growth on parts involved. Results of this program were presented by Professor F. G. Straub,¹ who carried out the investigation as directed by the author's company under its Utilities Research Commission.

Because valve stems and bushings had to have extremely hard, wear-resisting and non-galling surfaces to function properly for many years, a nitrided finish produced on the base materials a surface that had the desired properties. However, as was discovered during the research and only scantily mentioned in the very limited literature concerning nitrided surfaces, and as theorized by the author, such surfaces were far from corrosion resistant. High temperature steam caused nitrided surfaces to oxidize and this resulting in growth of the parts and subsequent seizure.

Tests Show Serious Consequences

Figures 1 and 2 give some data developed early in the research program and show why this oxidation was of such serious consequence. Of many tests run some showed effects of nitriding on alloys other than Nitralloy,* while others determined which alloys might be acceptable from an oxidation standpoint, and still others proved that the reaction was a steam-iron oxidation phenomena. Others involved spray welded carbide coatings and plated oversurfaces. In all, several hundred tests were made at temperatures ranging from 900 to 1200 F on some fifteen different alloys with five surface treatments. Data proved the cause to turbine manufacturers and offered some remedies. The problem was not, and still is not, a simple one. Various turbine manufacturers have test programs under way which may in the next few years produce a solution.

Turbine Blade Failures

Another problem some years past was the failure of a certain alloy turbine

* Nitralloy Type G, the Nitralloy Corporation: carbon, 0.3 to 0.4%; chromium, 0.9 to 1.5%; nickel, 0.5% maximum; molybdenum, 0.16 to 0.25%; phosphorous, 0.045% maximum; sulfur, 0.040% maximum; silicon, 0.2 to 0.4%; manganese, 0.4 to 0.7% and aluminum, 0.85% maximum.



Figure 4—Erosion-corrosion on casing of a turbine adjacent to the roots of the stationary blading.

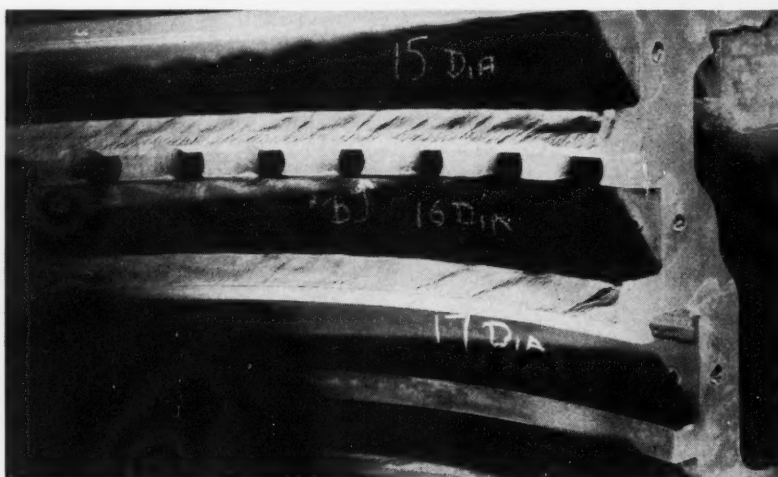


Figure 5—Erosion-corrosion on diaphragm of turbine.

blades which became so pronounced and so expensive that considerable effort was expended to reach a solution. The producer of this alloy believed the failures were caused by corrosion-fatigue, the corrosion being caused by carry-over of sodium hydroxide from the boiler water and deposition from steam onto the turbine blades and that the concentrated sodium hydroxide solution there deposited caused intergranular corrosion of the turbine blades.

Commonwealth Edison Company's extensive test work showed that (1) sodium hydroxide carryover did not occur and (2) that sodium hydroxide, under existent conditions, would not reduce the fatigue strength of the alloy. The material (probably because it did not dampen oscillations rapidly enough) had a fatigue limit too low for the service and failures were occurring on a service basis. Substitution of another alloy cured the failures.

Apparently all problems are not due to corrosion even though initial evidence may point that way. It often seems as

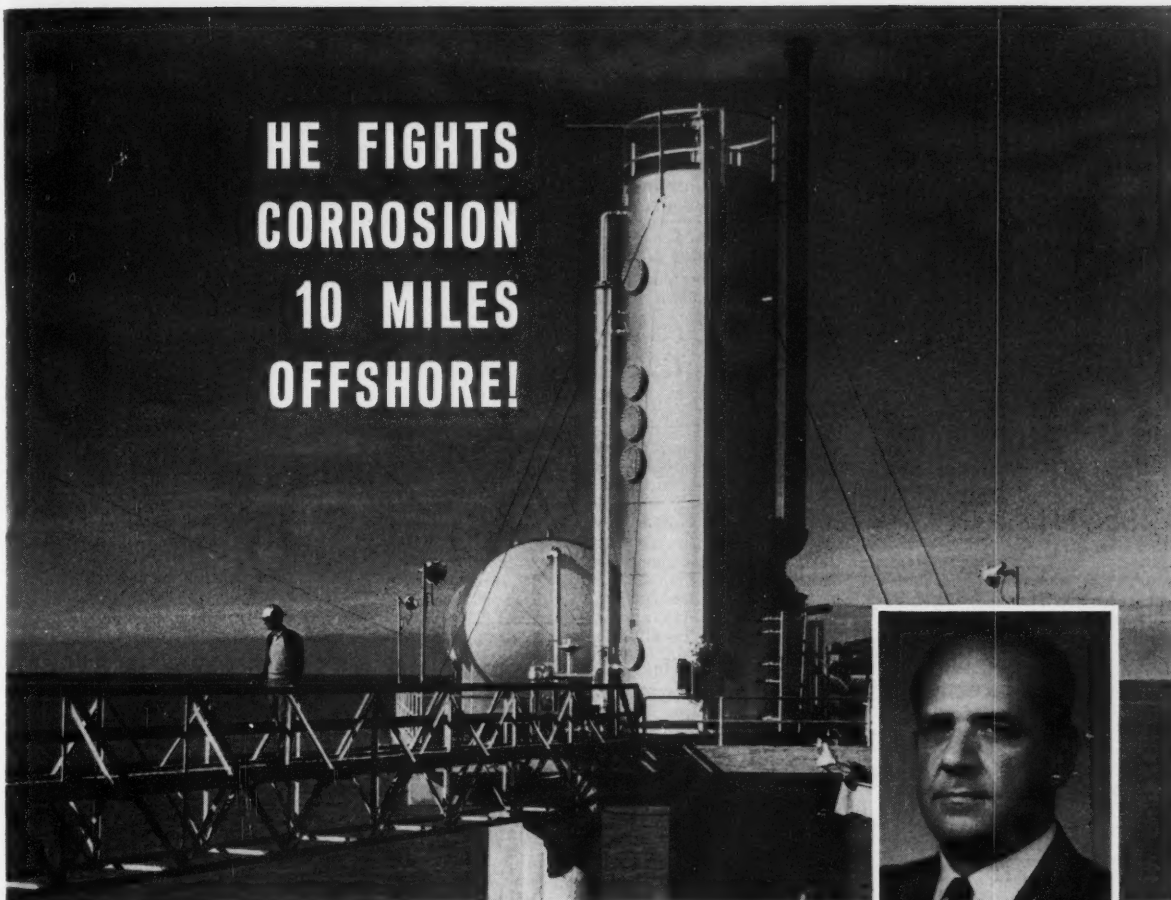
difficult to prove some contentions as it is to disprove others, or vice versa.

Boiler Environment Is Complex

The environment in the turbine and the condenser consists of more than steam, or steam-water mixtures but also includes some so-called noncondensable gases. Of these neither oxygen nor nitrogen are ever entirely absent. Carbon dioxide also may be present, depending upon the method of producing make-up water or upon infiltration of raw water. Sulfur dioxide is present in minute quantities as a result of decomposition of a portion of the sodium sulfite; hydrogen sulfide also may be present in minute amounts. Morpholine is present by choice and ammonia also is present, either as the result of decomposition of morpholine, from evaporated make-up or from raw water leakage. Hydrogen is present also, in the part per billion range, as a result of steam, morpholine and ammonia decomposition but primarily as the result of

(Continued on Page 22)

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Central Power—

(Continued from Page 20)

the very small and continuing corrosion of boiler metal.

Methane occasionally has been found, probably as a result of organic material decomposition which may have entered with make-up water or as thermal decomposition of morpholine. Of the above gases, only nitrogen, hydrogen and methane do not seem to play any part in affecting the corrosion of turbine and condenser components.

Steam-water mixtures, encountered first in the low temperature end of the turbine, travel at high velocities and are wear-causing. While there is not much doubt that this mixture is erosive in the absence of corrosion, there is no doubt that it is much more aggressive in removing corrosion products than it is in removing virgin metal.

Corrosion-Erosion in Turbines

Figure 3 is a shadow graph of a turbine blade from one of the last two or three rows of blades at the exhaust end of a large condensing turbine. The roughened portion represents metal loss caused by mechanical erosion of area where Stellite facing is used because of its wear-resistant properties. Figures 4 and 5 show the corrosion-erosion that occurred in the casing of a turbine adjacent to roots of the stationary blading. This condition was limited to the area where steam and water both were present and is many times more pronounced than similar destruction in turbines when corrosion is kept at a minimum by proper pH control. Corrosive gases which produced the accelerated attack were oxygen, carbon dioxide and sulfur dioxide and pH values were as low as 4 at times.

Metal removed from these areas was "recovered" on turbine blades farther along in the turbine, on condenser tubes and in the boilers. Repairs damage such as this are expensive and time consuming and the "recovered" metal is detrimental to operation. Iron loss at such low pH values is many times as great as that encountered when pH is controlled at about 9.

pH Adjusting Alkaline Natural

A pH adjusting alkaline material seems to be a necessity if corrosion is to be controlled in areas where steam-water mixtures occur as well as in condensate and boiler feedwater. Morpholine was selected for this purpose because (1) it is stable thermally, decomposition being very low at temperatures below 1100 F, (2) it is more soluble in the water phase of a steam-water mixture at saturation conditions than in the steam phase by a ratio of approximately 2.5 to 1 and (3) because of its water phase solubility, it is more effective than ammonia or other amines which might be used. When proper pH values are maintained, iron and copper corrosion products are readily kept below 10 to 20 parts per billion in the water returned to the boiler.

Some Problems With Condensers

Condenser tubes in various Consolidated Edison stations normally are arsenical or phosphorized admiralty metal. The tubes are rolled into Muntz metal tube sheets and the whole enclosed in cast iron or steel condenser shells. One condenser was tubed with aluminum tubes rolled into steel tube sheets.

A condenser on a recently installed unit contains over 200 tons of admiralty brass tubing and consists of almost 170 miles of tubes having a surface area of almost 217,800 square feet.

Mild corrosion of this large volume of metal in itself would be serious enough, but addition of the dissolved metal to the water in a high pressure boiler would cause more serious difficulties. To prevent this from happening, pH values are raised to about 9.

Effect of Gas Concentrations

If the steam-water mixtures in condensers always remained at saturation temperatures, no other remedy would be required. However, in some condenser sections the water is cooled below the corresponding saturation temperature, and this condition is most prevalent in the "air removal" sections through which the noncondensable gases flow as they are pumped out of the condenser. This sub-cooling, normally referred to as refrigeration, is quite small in some condensers while in others it is large enough so that several noncondensable gases may go into solution in large enough amounts to be troublesome.

Ammonia and sulfur dioxide both tend to concentrate in the cooled water and can cause corrosion, the sulfur dioxide by making the solution acidic and ammonia by causing it to be alkaline. Sulfur dioxide concentrations normally are considerably lower than are ammonia and, because they are almost always both present, ammonia is the governing component. Oxygen which is always present adds to the corrosion.

In one case, when ammonia concentration in steam entering the turbine was 0.15 to 0.25 ppm, the concentration in water removed from the surface of the tubes in the air removal section was several hundred parts per million, and its pH was 10.5. Copper solubility was very high and tube failures were experienced. Figure 6 shows the pH concentration effect of ammonia and the solubility of copper as a pH-dependent function. It is to be noted that ammonia concentrations were never high enough to cause the stress-corrosion effect, which results in tube failure by cracking.

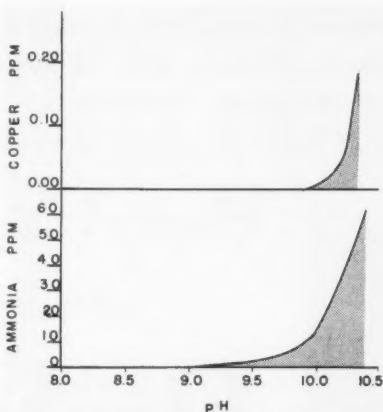


Figure 6—Copper solubility in presence of oxygen, shown in top graph. Effect of ammonia concentration on pH of pure water, shown in bottom graph.

Solution Still Is Elusive

A solution to this concentration of ammonia has not been found. The condenser manufacturer has recommended

removal of a portion of the tubes in this air removal section, thus reducing the sub-cooling effect and tending to reduce ammonia content of the condensed steam. Although this recommendation was followed there are insufficient data so far to determine how much improvement has resulted.

pH Adjusting Addition Required

Some sort of pH adjusting material seems to be required and in the authority's opinion will be necessary for many more years. So, it seems logical to use a metal other than a copper alloy in the air removal sections of future condensers. Because Types 304 or 316 stainless steels are resistant to the pH effect described they are being specified as replacements in air removal sections of existing condensers and will be specified in the future for this service.

There also is some loss of tube metal by erosion, because the high velocity steam-water mixtures act in a manner similar to that described for turbine blades. Until quite recently this has not been of much consequence, but now is causing some alarming losses.

Tests Made of Aluminum Tubes

Several years ago it was considered economical to use aluminum tubes in the condenser of a 325 MW turbine-generator. Corrosion tests on aluminum alloys indicated they would stand up satisfactorily and some installations were in service in other utilities. One unanswered question was whether or not they would perform adequately, as far as abrasion- or wear-resistance was concerned, when subjected to the impact of steam-water mixtures. The aluminum tube manufacturer indicated that the aluminum materials should be satisfactory. Tests on which this opinion was based did not simulate the contemplated service but rather were strictly for mechanical wear or abrasion resistance. The tests also indicated no appreciable difference in this property between a 6061 and a 3003 alloy even though the latter was softer and had a lower yield strength. Because of this and because the 6061 alloy was more expensive the 3003 alloy was selected for tubes one gauge thicker than was suggested for the 6061 alloy.

Tube losses due to impingement attack by the high velocity steam-water mixtures was so severe that this installation was abandoned in less than three years of operation at great expense. Another trial installation of some 60 aluminum tubes in another condenser on a different cooling water source was made primarily to determine the effect of this cooling water on the 3003 and 6061 aluminum alloys. Tubes were lost due to steam-water impingement, but this time comparisons of the resistance of not only the two aluminum alloys but also that of the admiralty brass tubes in this condenser were made.

The softer 3003 alloy had an average life of some 3500 hours in this environment while the 6061 aluminum alloy lasted 5500 hours. This showed that in this service the 6061 alloy would be serviceable 1.6 times as long as the 3003 material.

But the comparison between the aluminum tubes and the admiralty brass tubes is astounding. The brass tubes removed to make room for the aluminum tubes already had been in service eight years and were in almost perfect condition. Tubes in adjacent areas still

(Continued on Page 24)

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(Continued from Page 22)

are in excellent condition and there is no reason to believe they will not last for over 30 years. The conclusion is therefore, under these conditions resistance to steam-water impingement of admiralty brass tubes is at least 30 times as good as that of the aluminum tubes, and there is ample reason to believe the ratio is probably 100 to one.

It is postulated that the steam-water impingement attack of the aluminum tubes is a combination of corrosion and erosion in which the thin oxide formed on the aluminum is readily abraded away.

This reiterates the need for a true environmental comparison before proper selection can be made. In this case even

a proper screening between alloys was not possible until an environmental test was made.

Observations have been made on over 60 turbines and their associated condensers during the past 25 years. Many corrosion problems have been solved by proper practices, others are capable of solution while still others are considered to be normal and are tolerated. Routine maintenance restores the damaged parts.

In spite of the many possible effects on steam turbines and condensers, they have been relatively trouble free and probably are the most dependable components in central power station use.

Reference

1. F. G. Straub. Blue Blush Characteristics. Presented at the 17th Annual Meeting of the American Power Conference, March 30-April 1, 1958.

Technical Topics Scheduled for August

The following three articles will be included among the Technical Topics for August.

Cathodic Protection of A Buried Aluminum Pipe Line

Stress Corrosion Cracking of Stainless Steel Bolts in Water Environment

Reducing Corrosion of Power Plant Condenser Tubing With Ferrous Sulfate

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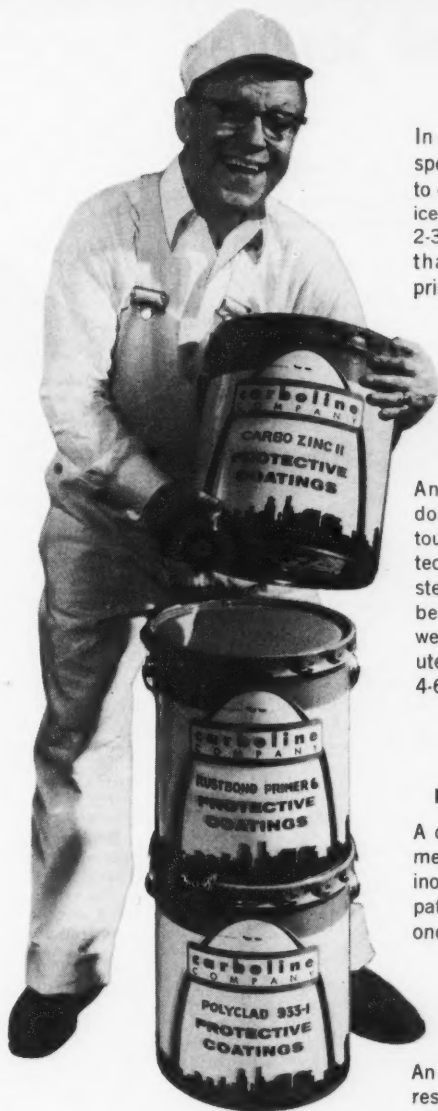
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Fluorocarbon Resin Piston Rings and Overbraided Hose Give Good Service*

L. A. Ferris

E. I. du Pont de Nemours & Company
Wilmington, Delaware

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temperatures. Their resistance to chemicals is less important in these specific applications.

Because of their ability to handle such difficult process gases as chlorine, oxygen and hydrogen, non-lubricated compressors are of particular interest to the chemical industries. These compressors do not contaminate the process gas with oil and often are used to handle air, nitrogen, ammonia, steam and organics such as methane, ethane, ethylene and hydrogen sulfide.

A variety of materials such as carbon-graphite and various resin bonded fabric laminates have been used, but limited life of the rings has been a major deterrent to the use of non-lubricated compressors.

Piston Ring Development

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load was reduced by a factor as high as 15 to 1; thermal expansion was cut in half. Of greater significance, however, was the increase of wear resistance by a factor as high as 500 to 1. Although the coefficient of friction of these blended resins is higher than of pure TFE resin, it is still at a satisfactory low level.

Bearings made of these blended resins prevent freezing and chattering because their starting friction is about the same as the running friction. Of course, these same mechanical properties are also desirable for piston rings.

In tests conducted by Koppers Co., Inc., and France Packing Co., Philadelphia, Pa., TFE resin blended with glass showed excellent wear resistance at room temperature and 400 F. Rings made of carbon-graphite showed similar wear resistance, but these rings have extreme brittleness and rigidity. When a carbon ring fails, it may cause scoring of the cylinder. Failures of TFE-fluorocarbon rings have resulted in no damage to the compressor.

Another advantage of piston rings made of the resins is elimination of the time consuming run-in followed by disassembly of the compressor to rotate the rings when carbon rings are used.

Case histories of compressors with rings made of resins include such services as air, dry nitrogen, carbon dioxide and pump services on hydraulic fluid and cold liquid oxygen. These cases included rings to 15 inches in diameter with piston speeds to 1800 feet per minute, pressures to 2400 psi and temperatures ranging from -320 to 350 F.

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Cost factors also show some of the advantages of piston rings made of TFE-fluorocarbon resins. The high cost of the rings themselves, as expected in a new product, is offset by elimination of breaking during installation, reduction in piston costs because less critical tolerances are possible and use of less expensive metals in compressor cylinders because the resins run best against ordinary cast iron rather than against chrome plated, nitrided or stainless steel cylinders or liners. The TFE rings also eliminate investments for lubricators and oil separators.

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To qualify for military specifications for high temperature assemblies, TFE resin hose must pass tests which include temperatures from -67 F to 450 F, pressures to 3000 psi, vibration test of 33 million cycles and combinations of these conditions in the presence of hydraulic and test fluids.

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Another case history involves a clothing manufacturer who was plagued with hose life averaging less than 30 days even though the hose was subjected to only 25 psi at 250 F. After test pieces of TFE-fluorocarbon hose remained in near perfect condition after six months'

Figure 2—Overbraid reinforced flexible hose made of TFE-fluorocarbon resins. Swage type coupling is shown in upper portion of photograph.

Figure 3—Rubber tire mold in which four swivel joints on the rigid piping are used to carry steam or superheated water under pressure to inflate a bladder during the curing operation. TFE reinforced hose is being used to replace the rigid piping which has frequent leaks.

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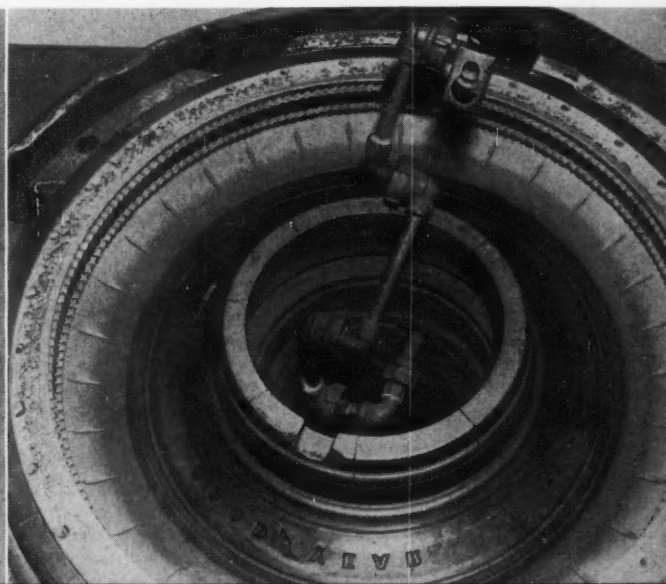
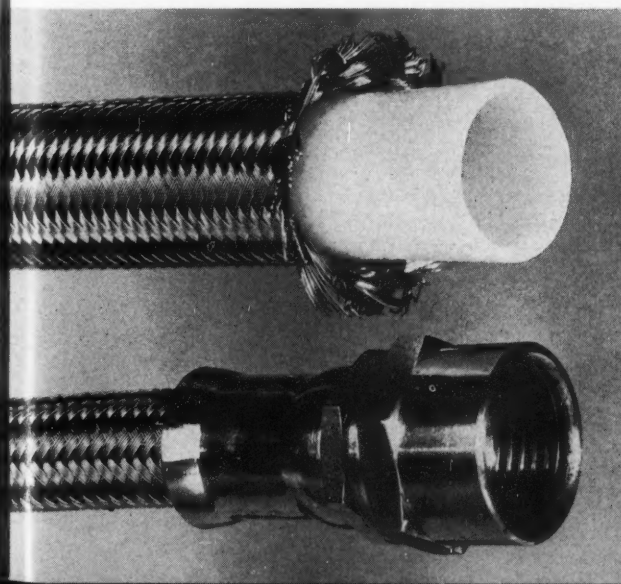
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Figure 1—Small piston ring of reinforced TFE-fluorocarbon resin. Stepjoint and metallic expander spring are shown. Rings of this material have been made in sizes to 29 inches in diameter.

temperatures. Their resistance to chemicals is less important in these specific applications.

Because of their ability to handle such difficult process gases as chlorine, oxygen and hydrogen, non-lubricated compressors are of particular interest to the chemical industries. These compressors do not contaminate the process gas with oil and often are used to handle air, nitrogen, ammonia, steam and organics such as methane, ethane, ethylene and hydrogen sulfide.

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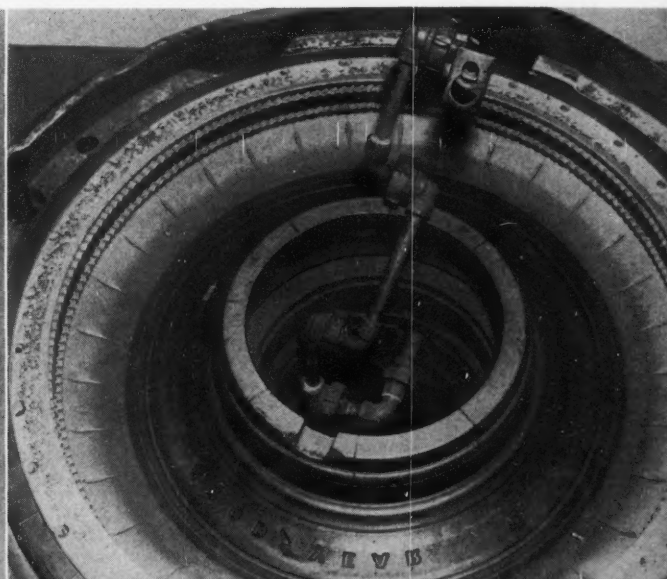
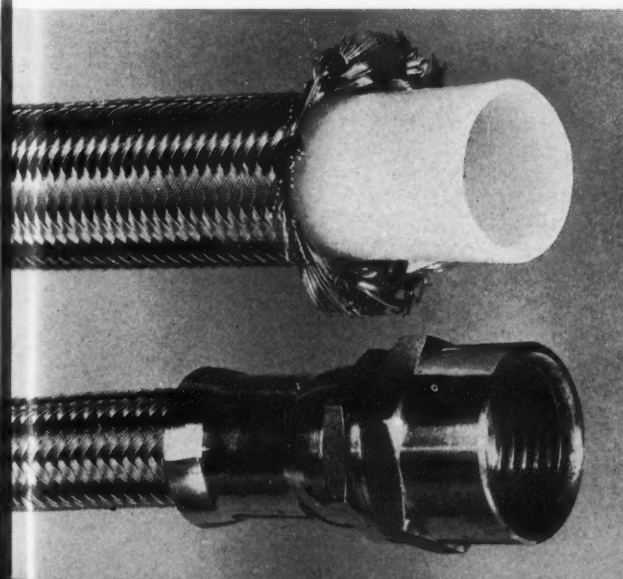
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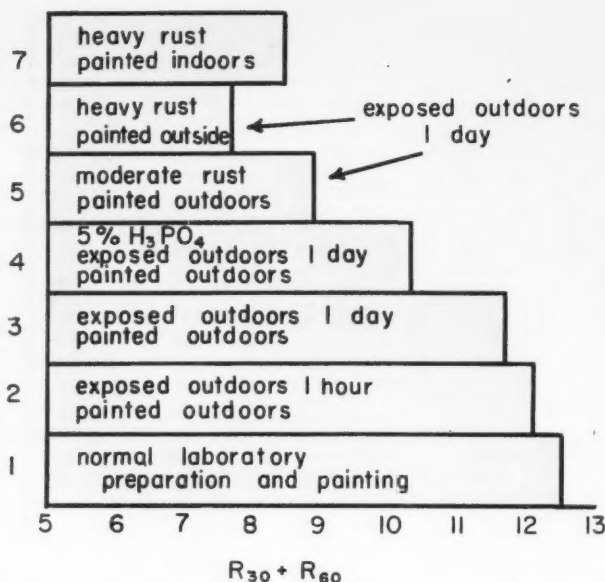


Figure 1—Effect of humidity, rust and 5 percent phosphoric acid wash on performance of coating.

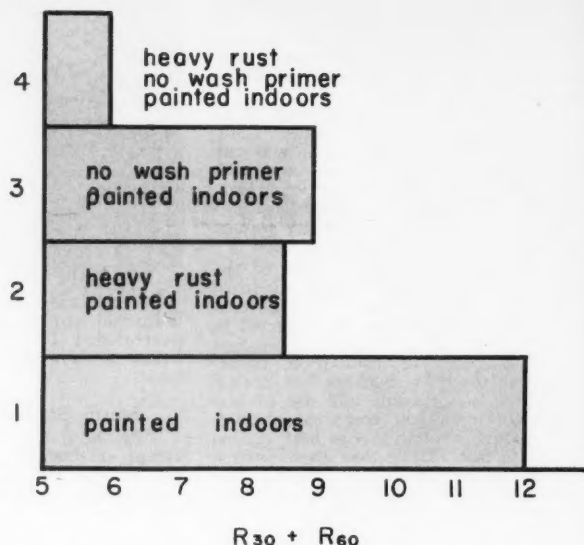


Figure 2—Performance of a bituminous coating system with and without wash primer on clean and rusty steel.

Laboratory Tests Give Rapid Evaluation of Ship Bottom Coatings*

J. R. Brown
Pacific Naval Laboratory
Esquimalt, British Columbia
Canada

Abstract

Discusses superior results obtained from laboratory evaluation of ship bottom coatings as compared with ship trial tests. Explains value of rapid evaluation, easy replication and controlled conditions which are possible with lab tests but not on ship trials. Describes tests conducted to study drydock variables, wash primer for pretreatment and influence of coatings on pitting rate of protected steel. 2.3.2

Introduction

LABORATORY EVALUATION of ship bottom coatings is looked upon with disfavor because it takes place under ideal conditions while service application takes place under many conditions other than ideal. However, laboratory evaluation compares much more favorably with ship trials than is generally conceded.

In practice, a single lab trial is like a ship trial: both are conducted under only one set of conditions. They differ in that the conditions are known for the lab trial and unknown for a service trial. In the lab these conditions can be varied so that the effect of a variety of surface preparation and weather conditions can be evaluated. In service a sufficient number of trials would have to be made to ensure that the entire range of service conditions were encompassed, thus re-

quiring that many ships be involved in the tests.

Laboratory testing also permits rapid evaluation without accelerating the rate of coating breakdown. Instead, sensitivity is improved for measuring the breakdown rate. For description of this evaluation, see author's article "Evaluation of Protective Coatings for Ship Bottoms," *Corrosion*, 15, 315t (1959) June.

Study of Drydock Variables

Test results obtained with a single coating system applied under a variety of conditions are presented graphically in Figure 1. The system consisted of wash primer pre-treatment, two coats of anti-corrosive and one of anti-fouling. Treatment started in all cases with wet sandblasting. This was followed by various treatments to simulate the effect of drydock exposure before painting: production of various degrees of rusting, treatment with 5 percent solution of H_3PO_4 and exposure for 24 hours to rain and high humidity. Temperature during the exposure and painting was about 50 F

and the relative humidity 70 to 100 percent.

The first bar in Figure 1 represents performance from an application made under ideal conditions. Distance between the end of the bar and dotted line represents the range of values obtained through many repetitions and so give a measure of the method's precision. Bar 2 involved exposure of the panels to high humidity outdoors for one hour before painting and then painting outdoors. Bar 3 is similar to the second except that the exposure was 24 hours—long enough for formation of a slight rust bloom. It can be seen that when application is made on clean steel, high humidity has little or no effect on performance of this coating system.

The difference a 5 percent H_3PO_4 wash makes under these conditions is shown by Bar 4. This treatment, which is supposed to inhibit rusting, actually results in poorer performance than no treatment at all. Previous work has shown that, under the low humidity conditions of

* Revision of a paper titled "Paints for Ship Bottoms—Case for Laboratory Evaluation" presented at a meeting of the Western Division, Canadian Region, National Association of Corrosion Engineers, Vancouver, British Columbia, February 10, 1960.

the laboratory, this treatment does not alter performance. The reason for this reduction may be that the phosphate salts, which are hygroscopic, absorb moisture under high humidity conditions.

Also shown here are the effect of several degrees of rusting when painting is carried out under high and low humidity conditions. The reduction in performance is as could be expected. Bars 5 and 6 represent the effect of moderate and heavy rusting respectively when application is made under high humidity conditions. Bar 7 shows the effect of heavy rust when painting is done under low humidity conditions. Comparison of Bars 6 and 7 shows that the detrimental effect of the rust layer is aggravated by high humidity, possible as a result of moisture absorption by the rust layer.

Thus, high humidity is detrimental to the performance of this coating system only when there is foreign matter on the surface to be painted—a good argument for improving standards of surface preparation.

Study of Wash Primer for Pre-Treatment

Figure 2 shows the effect of wash primer on this coating system. Bar 1 is for the system including wash primer and Bar 3 without. Corresponding performances for the same treatments applied over heavy rust are given by Bars 2 and

4. Value of the wash primer is evident for clean steel and rusty steel. This result is particularly interesting because there seems to be a general feeling that wash primer is more sensitive to surface preparation than normal primers. At least in this example, sensitivity of the wash primer was about the same as that of the bituminous primer.

A special tunnel (see Figure 3) was used to determine the effect of high velocity on coated panels. The tunnel subjects panels to water flow equivalent to ship speeds of 15 to 24 knots. It also has provision for cathodic protection of the panels.

The effect of high velocity on panels is shown in Figures 4, 5 and 6. These

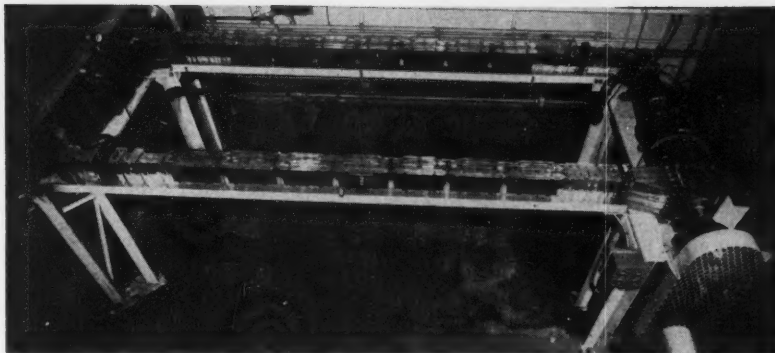


Figure 3—Tunnel used to expose coated panels to water velocity effects.

panels were under cathodic protection in the test tunnel for about 200 days. For most of this period, the potential was 1.0 volts to silver-silver chloride, but for the latter 70 days it was 1.2 volts. Figure 4 shows a bituminous system over wash primer that blistered badly near the score mark. Some blisters were swept away by the sea water. Figure 5 shows another bituminous system over wash primer which was more severely blistered and suffered considerable flaking.

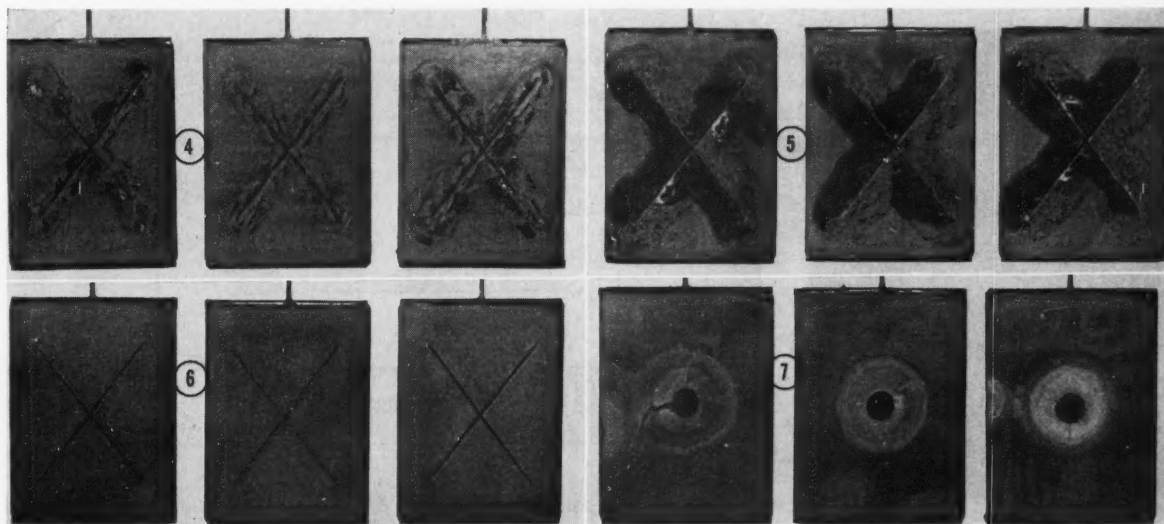
The vinyl system in Figure 6 appears unscathed by its exposure. However, this is not the case. Adhesion was reduced to nil for a good distance beyond the score mark apparently as a result of the complete destruction of the wash primer. Figure 7 shows the extent of this effect. Instead of a score mark, these panels had a 1/2-inch diameter window. However, the effect was the same. The reason for the unsymmetrical patterns on the two outer panels is that they were damaged at the edge during installation in the tunnel. For the first half-inch beyond the window, the wash primer was reduced to a mush; from there to

the edge of the paint, film strength was greatly reduced. Beyond that point, adhesion was excellent. On the panels coated with bituminous systems, though blistering and flaking occurred, the wash primer was relatively unaffected and retained its good adhesion.

This finding is interesting because, though the vinyl system makes a good bottom coating, it suffers from loss of adhesion and stripping. This stripping of vinyl paints in service is generally attributed to inadequacies in surface preparation even though, in some cases, special attention was given to this vital step. As a result, the vinyl system is said to be especially sensitive to surface preparation.

Wash primer in conjunction with a bituminous system is not especially sensitive to substrate surface condition. It has also been shown that wash primer undergoes degradation under cathodic protection when coated with vinyl red lead, but not when coated with a bituminous system. From these findings the inference can be drawn that stripping of vinyls (Continued on Page 30)

Figure 4—Blistering and flaking of a scored bituminous system under cathodic protection and high water velocity. Figure 5—Blistering and flaking of a scored bituminous system under cathodic protection and high water velocity. Figure 6—Panels coated with a vinyl system were scored and exposed to high water velocity with cathodic protection. Figure 7—Vinyl coating system with 1/2-inch diameter window after exposure to cathodic protection and high velocity. Loose paint was removed after the exposure to show poor adhesion.



Ship Coatings—

(Continued from Page 29)

experienced in service is due, at least in part, to a cause other than imperfections in surface preparation and that service data has indicated an erroneous conclusion.

Influence of the Coating on Pitting Rate

It has been said more than once that "good" coatings can be dangerous. The reason given for this is that coated steel is cathodic to bare steel and therefore, if a coating is so good that it allows very few bare areas, these areas in effect will be surrounded by large cathodes and pitting will be very rapid. Because accelerated pitting does occur and because efforts of paint formulators are in the direction of increased film continuity, it is of interest to investigate this potentially serious contention.

Figure 8 shows a pit which penetrated at the rate of 180 mils per year, far in excess of normal pitting. Rough calculations based on the weight of metal dissolved and an arbitrary 0.5 volt potential indicate that resistance of the coating would have to be less than 6000 ohms to allow the required current to pass. Intact coatings on similar panels generally have resistances in excess of 10^7 ohms and never exhibit corrosion when the resistance is above 10^8 ohms. The current could not, then, have been provided through the paint film but presumably came from the rest of the bare area. In fact cathodic deposit was visible at the cathode sites.

To determine the effect of size and distribution on the pitting rate of uncoated areas, an experiment was conducted using sandblasted panels coated with one wash primer, two vinyl anti-corrosive, and one vinyl anti-fouling. Holidays were created in the panel coatings by sandblasting through templates to give the following five patterns: (1) one holiday, $\frac{1}{16}$ -inch diameter, (2) one holiday, $\frac{1}{2}$ -inch in diameter, (3) 64 holidays, $\frac{1}{16}$ -inch diameter separated by $\frac{1}{32}$ -inch on a square pattern, (4) 64 holidays, $\frac{1}{16}$ -inch diameter separated by $\frac{1}{16}$ -inch and distributed over both sides of the panel and (5) bare panels.



Figure 8—Accelerated pitting (180 mpy).

Panels were prepared in triplicate and exposed to slowly moving sea water for 100 days. Pitting results from Patterns 2, 3 and 4 are shown in Figure 9. Results for maximum depth of pitting are given graphically in Figure 10.

In Figure 10 the three maximum values of pit depth obtained from the three samples of the same hole pattern are shown on each bar by the top and bottom of the cross-hatched area and the horizontal line within this area. It can be seen that the range of values for each pattern is sufficiently small compared to the results from the other patterns to indicate a significant dependence of maximum pit depth on bare area size and distribution. The smallest pit depth occurred with a single $\frac{1}{16}$ -inch diameter hole and was significantly less than that on the bare panels. If the coating acted as a cathode, the reverse would be the case. Increasing the size of the window to $\frac{1}{2}$ -inch resulted in increased depth of penetration; spreading this total area into 64 holidays of $\frac{1}{16}$ -inch diameter resulted in additional increase in maximum pit depth. Still more increase resulted from additional separation of the holes.

From these results, the conclusion can be made that coated steel is not an

effective cathode but that there is a relationship between size and distribution of holidays and pitting rate. Why this relationship exists is not established, but it may be a function of the availability of oxygen to the cathode areas. Normally, oxygen in a cathode plane must be supplied to a given incremental area through a cylinder in height equal to the thickness of the diffusion layer. However, an isolated cathode can draw oxygen from adjacent areas as well as from directly in front so that, in the extreme case of a point cathode, oxygen is drawn from a hemispherical volume and therefore is in much better supply than for the same point in a cathode plane. In any case, whether this is the best explanation or not, the phenomenon undoubtedly plays a contributing role in accelerated pitting.

Conclusion

As illustrated by these tests, laboratory evaluation compares more favorably with ship trials of coatings than is generally believed. The control offered in the lab and the relative ease of replication under varied conditions results in precision and speed not possible with coating tests conducted on ships. Consequently, the effects of many variables can be studied in lab tests, but these effects from ship trials can be only grounds for speculation.

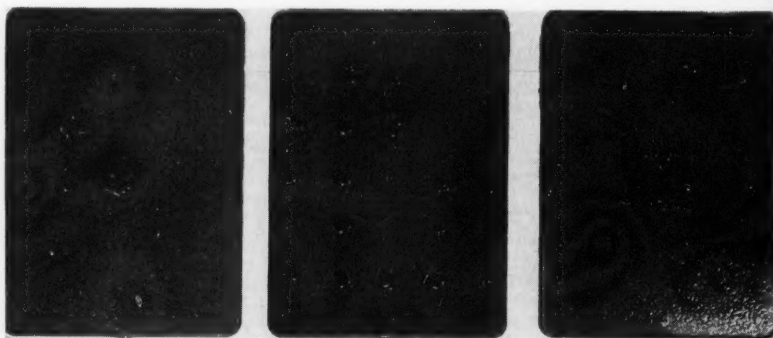


Figure 9—Three patterns of holidays sandblasted on panels to determine effect of coatings on pitting rates. Panels were subjected to slowly moving sea water for 100 days.

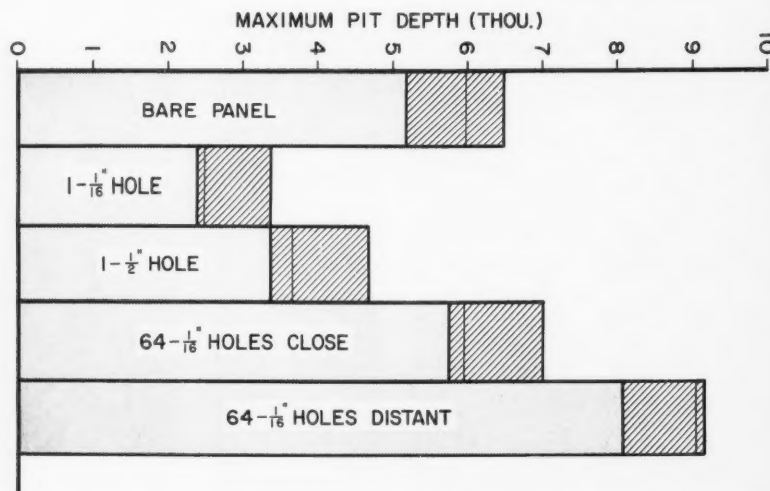


Figure 10—Graphic representation of maximum depths of pitting obtained on panels with various holiday patterns.

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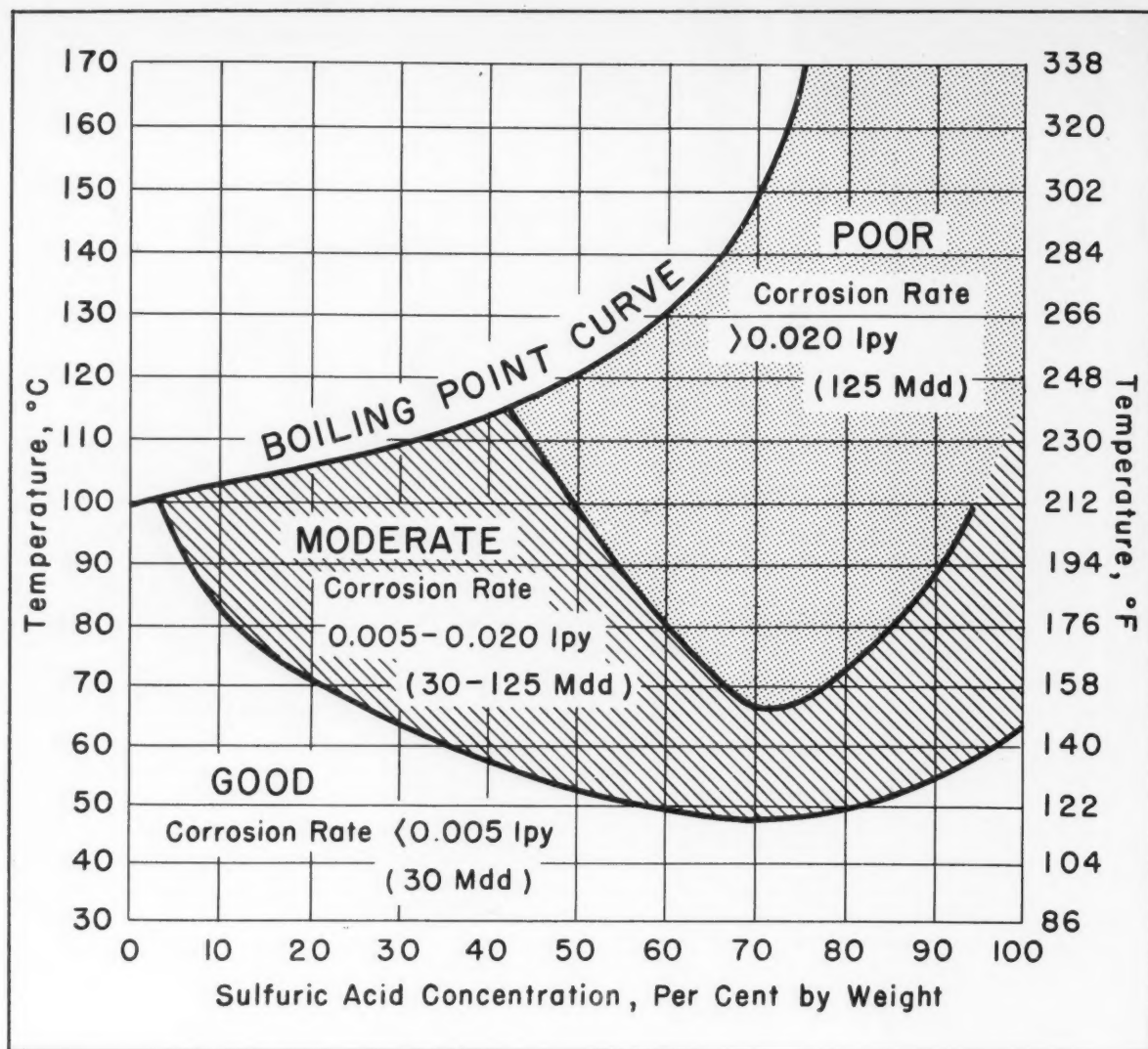
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*Plioweld is a trade name of The Goodyear Tire & Rubber Company.



Iso-Corrosion chart for Ni-o-nel alloy in sulfuric acid, based on laboratory data using pure acid.

How Ni-o-nel alloy resists attack by hot sulfuric acid

The few seconds it takes to read this chart may very well give you a practical answer to acids handling problems.

Ni-o-nel* nickel-iron-chromium alloy resists sulfuric acid solutions of up to about 40% concentration by weight at boiling temperature and up to 60% at 176°F. Moreover, it has good resistance to all concentrations at room temperature. The presence of oxidizing salts usually steps up corrosion resistance even more. That's why Ni-o-nel alloy is so suitable for mixtures containing nitric acid, cupric sulfate and ferric sulfate.

Readily welded. The metal-arc welding of Ni-o-nel alloy follows standard

welding procedures employing a "135" Ni-o-nel electrode. And, you exercise the same normal precautions about cleanliness of surface, use of annealed material, and removal of all slag before depositing successive beads in a multi-pass weld. Or you can use the tungsten-inert gas process equally well, with "65" Ni-o-nel filler metal.

To get detailed information on Ni-o-nel alloy, write for booklet T-37, "Engineering Properties of Ni-o-nel."

You'll get concrete facts on the remarkable corrosion resistance . . . the unique weldability for economical fabrication . . . of this alloy. For help in selecting materials to hold hard-to-handle corrosives, contact us. Our engineers will gladly provide the metallurgical information you want.

*Inco trademark

HUNTINGTON ALLOY PRODUCTS DIVISION
The International Nickel Company, Inc.
Huntington 17, West Virginia

NI-O-NEL®



Stainless Rotors Defy Corrosion of Wet Corn Milling

Precipitation hardenable stainless steel was used to cast impact rotors capable of withstanding the corrosion, abrasion and stress inherent in wet corn milling with centrifugal impact machines.

Fifty thousand pounds of steep corn per hour is fed into the rotor, which is rotating at 3600 rpm; the periphery of the 40-inch rotor is traveling over 500 feet per second. Kernels are hurled and smashed against impact posts along the rim of the rotor at velocities approaching that of a fired rifle bullet, according to the manufacturer of the machines, Entoleter, Inc., New Haven, Conn.

Highly corrosive corn steep liquor containing sulfurous acid is freed when the kernels are smashed and thrown from the rotor to impinge on the wall of the machine.

High stresses caused by rapid rotation and centrifugal accelerations of the rotors required design specifications of 100,000 psi tensile strength for the rotors.

The alloy selected for the rotors is type CB-7Cu with the composition C 16.5% Ni 4%, Cu 4% and CO5%. The alloy's tensile strength of 179,000 psi, yield point of 150,000 psi and elongation of 4% are developed in a duplex heat treatment of solution annealing by quenching after one hour at 1800-1850 F and precipitation hardening for one hour at 850-900 F with air cooling.

Stationary parts of the machine exposed to the wet corn slurry are cast from ACI type CF-8M stainless alloy; this alloy, which contains molybdenum additions of two to three percent, offers good resistance to the sulfuric acid present in the corn steep liquor.

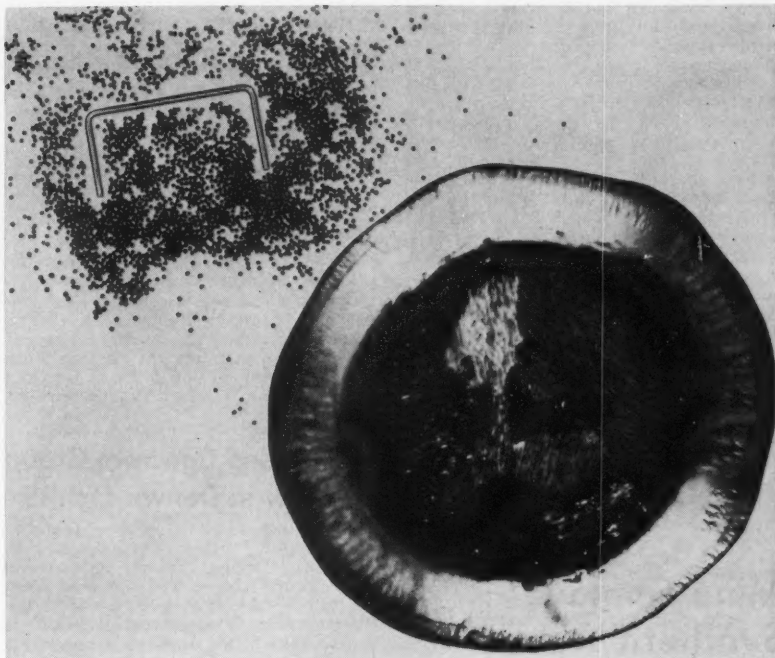
Three of these centrifugal machines have completed four years' service at a large corn milling plant with no downtime. They have increased starch yield three percent and cut processing costs by half a cent per bushel.

British Iron and Steel Group to Tour U. S. and Canada

Members of the Iron and Steel Institute, of Great Britain, will meet in the United States this fall at the invitation of the Metallurgical Society of AIME, 29 West 39th Street, New York 18, N. Y.

For three weeks, beginning October 19, the British group will tour industrial plants in Canada and the United States; they also have been invited to attend the National Metal Congress and Exposition of the American Society for Metals at Detroit October 23-26 and the Fall Meeting of the Metallurgical Society being held in conjunction with the Congress.

RECORD and REPORT



ATOMIC FUEL pressure spheres .001-inch diameter (upper left clustered around office staple) may provide the eagerly sought answer to economical, safe atomic power. Magnified 100 times at right, cross-section of single sphere shows uranium dicarbide center clad with impermeable pyrolytic graphite coating.

Atomic Spheres Promise Safe, Economical Steam Power

Atomic power produced from radioactive gases with existing high efficiency steam generating equipment coupled directly to an economical nuclear reactor is feasible, according to High Temperature Materials, Inc., 31 Antwerp Street, Boston 35, Mass.

The company has developed pyrolytic graphite pressure spheres to contain the radioactive gases which are by-products of the fission process. Each sand-size fuel particle is coated with the impervious pyrolytic graphite coating and the particles are then dispersed within 2½-inch graphite balls to attain near optimum fuel density. These fuel balls are loaded into a reactor and unloaded very simply.

The fact that pyrolytic graphite will not react with most available coolants affords significant construction and operating economies by allowing direct generation of uncontaminated steam. High temperature strength of the pyrolytic graphite coating allows operation at efficient reactor temperatures without dissipation of radioactive by-products. At 4500 F the pressure of fission gases to be contained within each fuel particles would reach 80,000 psi but

would require only 40 to 80 microns of pyrolytic graphite over a 100 to 300 micron diameter fuel particle.

Results of tests now in progress indicate fuel economy will also be improved by reducing costs of fuel reprocessing. Chemical reprocessing of metal clad fuel elements costs a minimum of \$56,000 a batch and is performed when the metal cladding begins to break down rather than when the fuel is exhausted. Reprocessing of pyrolytic graphite clad fuel may be accomplished by a simple crushing and gravity separation process at the end of the fuel's useful life. Further economies result from the spectroscopic purity of pyrolytically deposited graphite and the high neutron economy of graphite.

The integrity of the coated particles has been subject to rigorous testing, High Temperature Materials, Inc., reports. Particles have been cycled from room temperature to 3632 F repeatedly, followed by boiling in nitric acid. These tests have shown no increase in activity over background levels. Irradiation tests by the Atomic Energy Commission at the Oak Ridge National Laboratory also are being conducted.

Costly Titanium Fan Pays For Itself First Fifteen Weeks

A titanium fan installed to exhaust highly corrosive gases from a rotary furnace paid for itself the first 15 weeks of operation.

The furnace is used in salvaging 75,000 to 100,000 pounds of copper wire coated with polyvinyl per day. Combustion of the polyvinyl in the furnace creates gases containing ferric, cupric and hydrogen chlorides. These gases pass from the furnace through a salt water spray unit for cooling and scrubbing and the exhaust contains residual hydrochloric acid plus wet iron and copper salts.

The 49-inch titanium fan and its housing costs \$17,000. Carbon steel fans previously used survived about one week; each failure cost about \$1100 (material \$400, downtime \$600 and labor \$100).

The titanium fan was constructed of $\frac{1}{8}$ -inch commercially pure titanium sheet with a $\frac{5}{8}$ by 12-inch titanium hub. It weighs 200 pounds and is capable of exhausting 20,000 cubic feet of gases from the furnace per minute. The fan is in service at Nassau Smelting and Refining Company, Tottenville, Staten Island, N. Y., a division of Western Electric Company, Inc., 195 Broadway, New York 7, N. Y.

Mine Employs Synthetic Rubber For A-C Cables

Synthetic rubber connectors are used to join high voltage wires of the electrical system of one of the few coal mines in the United States using alternating current.

The connectors, rated at 7500 volts and permanently vulcanized to the cable jacket, have been in use for over a year and have shown good resistance to abrasion, moisture and aging. Made from Hypalon, a chlorosulfonated polyethylene developed by Du Pont, the connectors are manufactured by the Electrical Products Division of Joy Manufacturing Company, 1201 Macklind Avenue, St. Louis 77, Mo.

Alternating current was specified for the installation to gain economies both in initial installation and in operations and maintenance. Unlike direct current operations where high voltage is not carried beyond the standard rectifier set, rotary converter or motor generator, the alternating current installation carries high voltages well into the underground mine entries. Connectors are used not only where cable lengths meet but where circuits enter or leave control or transformer boxes or other operating equipment.

Safe high voltage connectors were fundamental in developing a safe alternating current electrical system for the mine. Joy engineers spent five years of research before they found an elastomer that could stand up to mine requirements.

Another synthetic rubber, Neoprene, is used for cable jacketing throughout the mine and for conveyor belts. Both Neoprene and Hypalon have good resistance to flame, a quality which is important in

preventing the spread of fire along conveyor belts and electric cables. They are produced by E. I. du Pont de Nemours & Company, Wilmington, Del.

The coal mine referred to is located near Parrish, Ala., and is owned by Southern Electric Generating Company, 600 No. 18th St., Birmingham 2, Ala.

Polymer (France) Building \$12 Million Rubber Plant

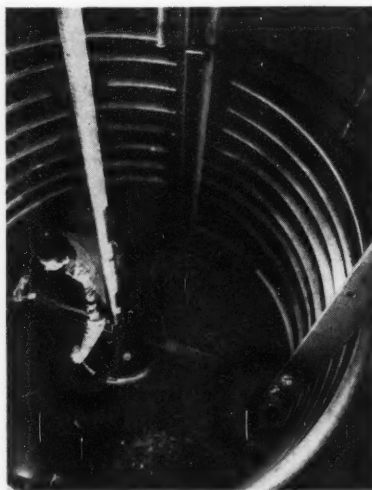
A contract for the design and construction of a \$12 million specialty rubber plant at Strasbourg, France has been let by Polymer Corporation (SAF), France, newly formed subsidiary of Polymer Corporation, Ltd., Sarnia, Ontario. The plant, scheduled for completion the last half of 1962, will have a capacity of 10,000 tons of specialty rubber per year. Construction will start this summer.

Badger (France) S. a. R. L., Paris, received the contract; Badger N. V., Dutch associate company of Badger (France) will assist in handling the project. Both companies are affiliated with Badger Manufacturing Company, Cambridge, Mass.

Prestressed Concrete Group To Meet in Denver Oct. 15

The Prestressed Concrete Institute will highlight "New Opportunities in Structural Design" during its national convention October 15-19 at two Denver hotels, the Brown Palace and the Cosmopolitan. The convention, which is the seventh in the Institute's history, is being held in cooperation with the University of Colorado.

Plans include speakers, seminars and social events. About 1,000 delegates, architects and engineers are expected to attend.



TITANIUM COOLING COIL in chlorinator tank resists highly corrosive hot wet chlorine. Coil controls heat given off when chlorine reacts with lime slurry in tank to produce calcium hypochlorite at Pennsalt Chemicals Corporation's Wyandotte, Mich., plant. Installed in 1959, the heat exchanger has cut downtime and shown no sign of corrosion. Build-up on coil from batch washes off easily.

PERIODICALS

Petrole Informations. (In French) 88 pages, $8\frac{1}{2} \times 10\frac{3}{4}$ inches. Published by Petrole Informations, 15 Rue Danielle-Casanova, Paris 1, France. For 22 issues, countries other than France, 100 NF.

Technical, industrial and economic information on the petroleum industry in France, Europe and the Mediterranean conclave.

Transactions of the Japan Institute of Metals. (In English) Vol. 1, No. 1, July, 1960. 68 pages, $8\frac{1}{4} \times 11\frac{3}{4}$ inches. Quarterly. Japan Institute of Metals, 69 Minamimachi, Sendai, Japan. Subscriptions, per year, \$5.

The Japan Institute of Metals, recognizing the language difficulties faced by scientists in using the data developed by the institute and published in Japanese in its official journals, is now publishing in English, French and German selected articles from the Japanese language periodical.

The first issue, printed on high quality coated book paper, includes articles on oxidation equilibria of uranium dioxides, plastic deformation of aluminum crystals, concentration of hydrogen ions on the Flade potential of nickel, equilibrium between dissolves oxygen in liquid iron and the hydrogen-water gas mixtures, and others.

The magazines also consider contributions from outside sources for publication. Instructions are included on the inside back cover of the first issue.

Current Engineering Practice. (In English) 38 pages, $8\frac{1}{2} \times 10\frac{3}{4}$ inches. The East Asiatic Co. (India) Pvt., Ltd. Bombay, India. Subscription, per year Rs. 25.00.

Consisting of technical articles in English on a wide variety of subjects from diverse sources. The September, 1960 issue (Vol. 3, No. 3) includes articles by American and British authors, includes product-oriented and general items of engineering news and a book review section. Subject matter includes lubrication, epoxies, electronic circuitry and others.

Global Technology. 56 pages, $8\frac{1}{4} \times 11\frac{1}{4}$, saddle stitched. Vol. 1, No. 1, April, 1961. \$25 a year. 2-year subscription, \$40. Trans-World Publishing Co., Euclid Ave. at East 107th St., Cleveland 6, Ohio.

A magazine consisting of several types of information: Short abstracts of technical articles referred to by descriptive phrases, but not referenced as to source; a listing of technical translations available through Office of Technical Services; summaries of technical articles (in this issue from Russian, Japanese, Swedish, French, German and other sources); original articles (in this issue a discussion of the problem of time relativity); a summary of information from technical exhibitions (German Hanover industrial fair).

Data are derived from surveys of 2000 technical publications in 35 languages and are comprehensive over the whole range of industrial activity. Photocopies of English translations articles referenced are offered at 75c per page.

The publication also offers to make a product search among advertisements

in all foreign publications on any specified subject, or restrict it to publications from specified countries. Photocopies of advertisements found are provided at a scale related to the dimensions of the search. A trial search producing 12 photocopies costs \$50. A three-month search with photocopies at the end of each month costs \$750. Photocopies include translated information as to manufacturer, country, address, performance data and other key information.

The publication also offers research, engineering research, foreign market explorations and foreign language translations.

Fluoro-Rubber Packing Resists Liquid Methane

A fluorinated synthetic rubber packing for swing joints used to load liquid methane, which is liquefied at -258°F , showed no sign of deterioration after 30 hours of loading. Plastic packings failed because of the low temperature at which methane passed through the swing joints.

The rubber packing, made of Kel-F elastomer, was developed by the Chiksan Company, Brea, Cal., for test loading of the tanker "Methane Pioneer" at Lake Charles, La., for shipment to Canvey Island, England, where it will be converted to gas.

Depleted gas reserves in Great Britain and Europe have prompted government and private gas suppliers to investigate the importation of liquid methane. The Sahara and ports along the North African coast appear to be the primary sources of methane.

Kansas State U. to Continue ASHRAE's Environment Studies

The study of man's reaction to various thermal and radiant environments carried on by the American Society of Heating, Refrigerating and Air-Conditioning Engineers 234 Fifth Avenue, New York, N. Y., during the past few years will be continued by Kansas State College of Agriculture and Applied Science, Manhattan, Kan.

The \$150,000 controlled environment room now housed in ASHRAE's laboratory at Cleveland, Ohio, will be moved to the college's new Environmental Research Center and operated under a co-operatively financed program to augment the work now being completed in Cleveland. An advisory panel composed of members appointed by the society and the college will assist the college staff in carrying out the program.

The transfer of the environment room to the college is the first major decision regarding the research activities of the society since it announced in December it was increasing grants to colleges and private laboratories and closing its laboratory.

The solar calorimeter used for many years to study solar heat gain through glass and other structural components and the influence of shading devices on heat gain will also be made available to

a university laboratory interested in research in this field.

The society reports that without a laboratory more committee work will be required to generate and monitor the increased number of projects in cooperative institutions and that future research will be aligned closely with industry problems.

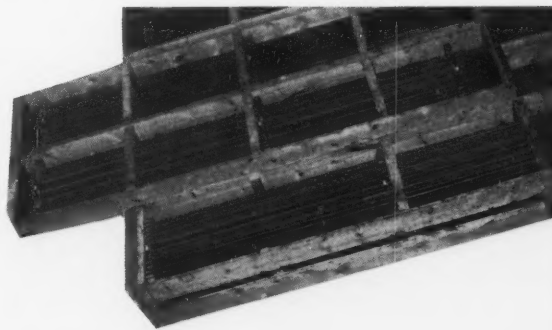


BRITISH METALLURGIST Bernarr E. Hopkins (right) who has been named to fill the chair of the new Battelle Professor of Metallurgy at Ohio State University during the 1961-62 academic year is shown in London with Mars G. Fontana, ex-president of NACE and now chairman of Ohio State's department of metallurgical engineering, during Fontana's recent visit to attend the International Congress on Corrosion.

NACE's 18th Annual Conference and 1962 Corrosion Show will be held March 18-22, 1962, at the Municipal Auditorium in Kansas City, Mo.

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RUBBER HOSE loads pressurized cement under hot sandblasting conditions into barge from storage silo. Flying through hose are limestone, granulated slag and silica at 30 psi and often at 200 F as heat generated during manufacture is retained in mix during storage. Loading requires flexible hose to move with barge as it sinks during loading. Wire reinforced hose tested failed after two months at the Green Bag Cement Company, Neville Island, Pittsburgh, Pa., while Conventapipe rubber hose developed by B. F. Goodrich Company, Akron, Ohio, has handled a half-million barrels of cement during past 15 months.

Plastic Engineers Meeting To Highlight Design

"Plastics Revolutionize Design" is the theme of the Society of Plastics Engineers' 18th Annual Technical Conference to be held at the Penn-Sheraton Hotel in Pittsburgh, Pa., January 30 to February 2, 1962.

Highpoint of the conference will be a technical paper by Giulio Natta of Montecatini in Milan, Italy, entitled "Progress in Stereo-Regulated Polymers."

Technical areas to be covered by approximately one hundred papers include new plastics formulations such as polyolefins, urethanes and polystyrenes (processing, properties and applications), reinforced plastics (uses and new developments in testing) and plastics-metals combinations. Injection molding and extrusion, fast moving blow molding and thermo forming, and auxiliary plastics processing equipment will also be discussed. In addition, special sessions are scheduled for applications of plastics in construction and in space vehicles and missiles.

Rocket Fuels Information

Safety information on the manufacture, storage and transportation of rocket fuels will be assembled and made available to government agencies and their contractors by the Manufacturing Chemists' Association, Inc., 1825 Connecticut Avenue, N. W., Washington 9, D. C.

The newly formed Chemical Propellant Safety Subcommittee composed of representatives of a dozen chemical propellant manufacturers will carry on this work under the directions of the General Safety Committee. Non-petroleum liquid propellants will receive early attention by the group.

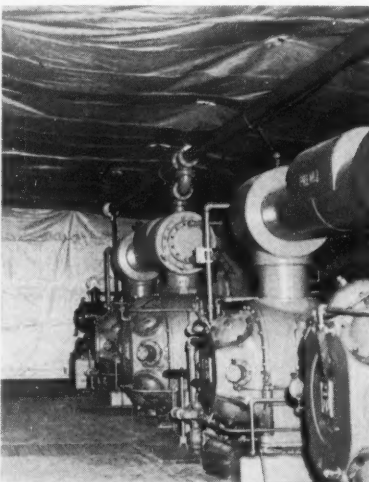
Epoxy Repairs Tidegate

A leaking tidegate of sewerage system of Jersey City, N. J., was repaired with epoxy resin in November, 1959. Rubber

gaskets had failed to withstand water pressure, abrasive action of floating objects and corrosive chemicals and waste materials.

Inspection a year later revealed the epoxy repairs to be leakproof. Epoxy resin compound used was Metalset A-4, made by Smooth-On Manufacturing Company, Jersey City, N. J.

Designed to regulate pressures caused by storm conditions or high tides, Jersey City's tidegate opens into Newark Bay to release excess storm water, closes to keep tide from entering the system.



PLASTIC COATED burlap tents limit humidity corrosion of mothballed Army Ordnance machinery in Louisiana. Suspended by steel cables inside warehouses, they provide localized areas for efficient operation of dehumidifying units. Ceilings and walls are 10-ounce burlap extrusion coated on both sides with a 4-mil film of Tenite polyethylene produced by Eastman Chemical Products, Inc., 260 Madison Avenue, New York 16, N. Y.

NASA Sponsors Research On Superfluid Helium

Superfluid helium research is being sponsored by the National Aeronautics and Space Administration, which has awarded Stevens Institute of Technology, Hoboken, N. J., a \$27,698 grant to make study.

The research will include verification of whether four or five million volts of electricity speeds up the flow of superfluid helium from one point to another and whether the superfluid's resistance to or pressure against other objects vanishes when its flow slows to almost immeasurable rates.

Superfluid helium is created by bringing helium to a temperature two degrees above absolute zero. Unlike any other material, superfluid helium not only does not freeze at any temperature except under pressure but transmits heat in waves. It is about 1200 times more conductive than copper.

Plastic Coated Paperboard Has High Resistance

Paperboard having resistance similar to Saran-type plastics to moisture, grease, air, gases, odors and many acids, alkalies and solvents is available to the packaging industry, according to its producer, Potlatch Forests, Inc., Lewiston, Idaho, which also plans to produce high barrier paper.

First uses for the paperboard are expected to be for bakery boxes, meat trays, picnic plates and cups.

Future packaging applications range from cookies to chemicals, from mustard to motor oils and from frozen foods to photographic film, Potlatch believes.

Called Daran, the coating is derived from a new polyvinylidene chloride emulsion developed by Dewey and Almy Chemical Division, W. R. Grace & Co., Lexington, Mass.

Catalog of Microcard Publications, 1961-62. 32 pages, 6 x 9 inches, paper. May 1961. Microcard Editions, Inc., 901 Twenty-Sixth St. N.W., Washington 7, D.C. FREE.

A list of the reprint material available on Microcards with the prices for cards.

List of Publications, April 1961. 62 pages plus Order Form Cover. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. FREE.

A comprehensive list of all ASTM standards collected and individual, together with prices and all necessary information for ordering copies. There is an alphabetical subject index.

Embrittlement by Liquid Metals. By William Rostoker, Joseph M. McCaughey and Harold Markus. November 18, 1960. Reinhold Publishing Corp., 430 Park Ave., New York 22, N.Y. Per copy, \$7.95.

An account of work done under sponsorship of the Chief of Ordnance, Department of the Army. The phenomena are described in an integrated and logical way, then the contemporary state

(Continued on Page 38)

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An ideal corrosion inhibitor for use in LPG product pipelines, Unicor LHS is soluble in all hydrocarbon liquids. It stays with the product and gives continuing protection even to the point of safeguarding your customer's equipment.

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BOOK NEWS

(Continued From Page 36)

of understanding on the mechanisms of fracture is reviewed. Experimental work is included.

Contents include: Literature review, occurrence of liquid metal embrittlement, crack propagation, factors influencing embrittlement by liquid metals, delayed failure by liquid metal embrittlement, theory of the fracture of metals

and mechanism of liquid metal embrittlement.

There is a brief subject index. Each section is thoroughly referenced and the book is liberally illustrated with appropriate diagrams and photographs.

Boilers: Types, Characteristics and Functions. By Carl D. Shields. Feb. 23, 1961. 559 pages, 6 7/8 x 10 inches, full cloth. F. W. Dodge Corp., 119 W. 40th St., New York 18, N. Y. Per copy \$15.

A comprehensive book on boiler technology covering most of the practical aspects of the subject. At least 45 references are found in the alphabetical subject index keyed to "corrosion," and other references are to related phenomena.

It is organized into 32 chapters in six major sections. The sections are: Classification, design, steam generating equipment, construction, regulation, status. For example, in the section on construction, sub-headings are: steel boiler manufacture, erection, settings, firing equipment and auxiliaries.

The 22-page index permits easy access to the information which is cast for the practical operator. The book has hundreds of photographs and diagrams related to the data in the text.

"Penetration of Plant Roots in Pipe Line Coatings based on Asphalt Bitumen" and "Glass Felt as a Coating for Steel Pipe. Jan., 1961. (In Dutch). 7 pages 8 x 11 1/2 inches. Netherlands Institute of Metals, Corrosion Committee. Postoffice Box 52, Delft, Holland. Per copy, F, 2.50.

English abstract of the first article indicate examination shows bitumen pipe line coatings are penetrated by roots,

and measures of combating them are described. Numerous tabulated data are given in the second article.

Conversion Factors and Tables, Third Edition. By O. T. Zimmerman and Irvin Lavine. 680 pages, 4 3/4 x 6 inches, Flexible binding. 1961. Industrial Research Service, Masonic Bldg., Dover, N. H. Per copy, \$7.50 (outside U.S. \$8.25).

Over 15,000 conversion factors and 132 pages of conversion tables based on latest and most accurate data. English and metric units, and factors for conversion of weights and measures in 95 foreign countries. There is an alphabetical subject index.

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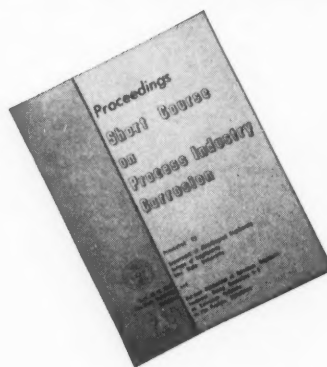
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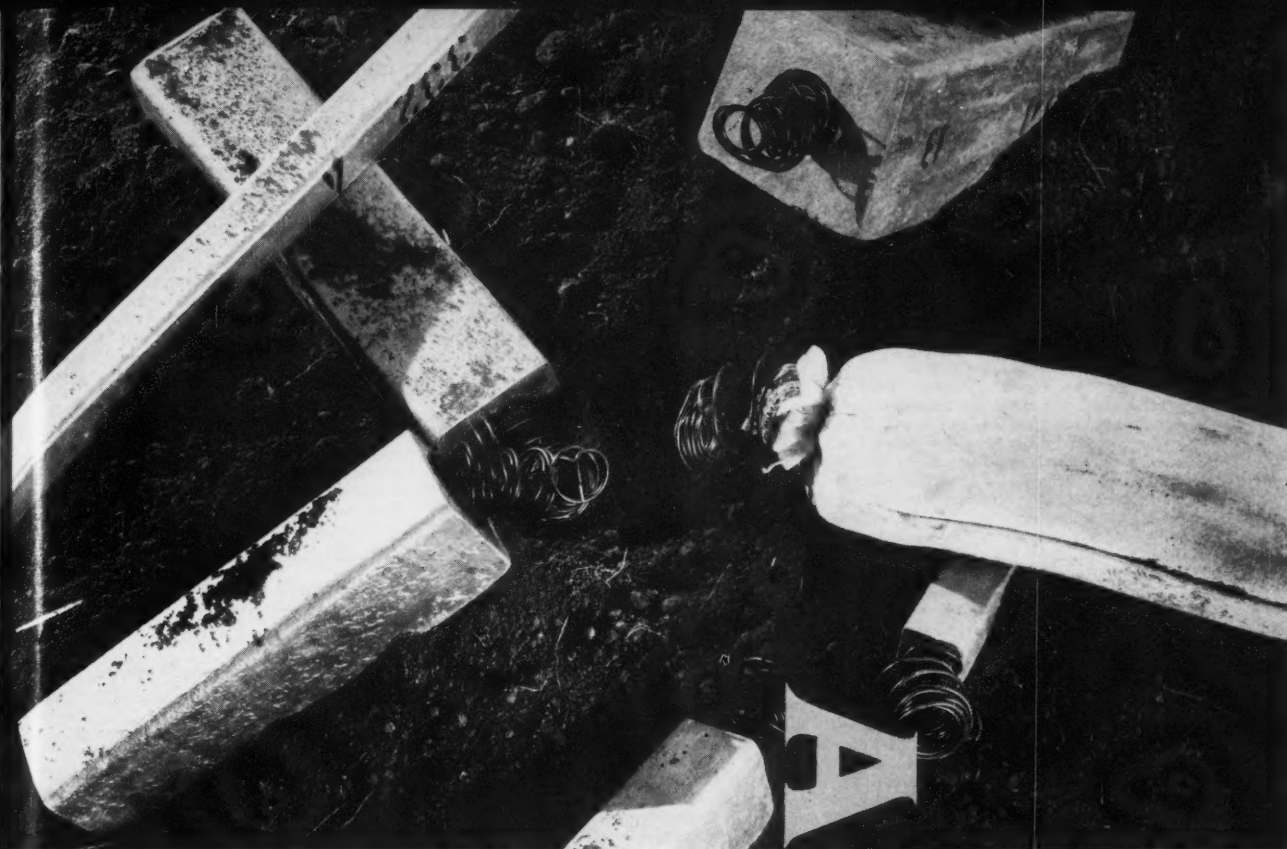
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These papers are by engineers concerned with controlling corrosion in some of the nation's largest chemical processing plants. Considered are use of metal and plastic materials in a wide variety of environments, including most of the common acids and alkalis; special problems found in both low and high temperature processes, corrosion measurements, coatings, design and other practical information.

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Equipment Services

NEW PRODUCTS

Materials Literature

Aluminum

Aluminum tubes are being used as containers for uranium oxide fuel pellets as part of the U. S. Atomic Energy Commission's program to develop economical organic cooled reactors. The tubes, fabricated from powder using the impact extrusion process, provide strength (up to 900 F), high thermal conductivity, low neutron absorption and resist corrosion of organic coolants. Terphenyl, a carbon-hydrogen compound, flows over the exterior of the tubes and acts as moderator to control the atomic fission process in the fuel pellets in the tubes, helping to sustain the chain reaction. The tubes are produced by Alcoa Aluminum Company of America, 1501 Alcoa Building, Pittsburgh 19, Pa.

Colloidal alumina capable of acting as an emulsifier and a thickening, dispersing and suspending agent in both water and water-alcohol systems has been developed by E. I. du Pont de Nemours and Company, Inc., Wilmington 98, Del. Called Baymal, it is a white, free flowing powder composed of clusters of minute fibrils of boehmite alumina. Dispersed in water, the fibrils assume a positive charge and when dried, interlace to form a positive charge film;

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ZINC SHIELD

Ferrous surfaces too large or critical for ordinary galvanizing techniques can be zinc-clad with this metallic zinc-copolymer coating for cathodic protection against filiform corrosion . . . a recent application being AN/FPS-35 Search Radar Antenna.

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Zinc Deposit — 1/4 oz. zinc metal/sq.-ft./coat (at 1200 sq. ft. gal. application rate)

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this process permits the film to affix itself to negative surfaces such as glass, paper and clay. Other properties of Baymal include high surface area and low toxicity. Also Du Pont claims it to be the most thixotropic material known to them.

Hard anodized coatings for imparting wear resistance to aluminum alloys are covered in a seven-page engineering report released by Allen Aircraft Products, Ravenna, Ohio. Report details physical properties and results of complete hardness studies and illustrates the effect of coating on part dimensions. Specific cost figures are given.

Cleaners

Dry, granular acid, easily transported and stored, for use in dissolving calcium carbonate and other acid soluble materials, including silicates and mud cakes, is described in a product data sheet from Dowell Division of Dow Chemical Company, Box 536, Tulsa, Okla. Called Instant Acid, it is packaged in 50-pound cartons. The mixing ratio is one pound per gallon of water.

A concentrated liquid detergent which removes grease and dirt from evaporator coils, air cooled condenser coils, metal air filters and fans is marketed by Calgon Company, Hagan Center, Pittsburgh 30, Pa., as CalClean. The company also markets a 3 1/2 gallon sprayer with a hand operated air pump and an adjustable spray nozzle, a 20-inch brass extension tube and 10 feet of hose. The detergent, which is mixed with water, is recommended by Calgon for painted and enameled equipment and for most metal, cement, glass, tile or linoleum surfaces.

Coatings, Inorganic

Zinc-filled ready-mixed coatings for structural steel, tanks, dam gates and other on-shore, off-shore installations are manufactured by Truscon Labora-

tories, 1700 Caniff, Detroit 11, Mich. Called Chem-Zinc, it self cures, according to the manufacturer, has unlimited storage life and is resistant to rain splash, turbulence and immersion in fresh or salt water minutes after application.

Galvanic principles of inorganic zinc protection in immersion and non-immersion service are described in a bulletin available from the Carboline Company, 32 Hanley Industrial Court, St. Louis 17, Mo.

Coatings, Metallic

A zinc-magnesium alloy (97 Zn 3 Mg) for hot dip galvanizing of steel is being marketed by Eagle-Picher Company, American Building, Cincinnati 1, Ohio. Six-year evaluation tests at Dow Chemical Company's Electrochemical Research Metal Cleaning Department in Freeport, Texas, indicate 20 to 90 percent improvement in corrosion resistance when magnesium is present in amounts of 0.04 to 0.05 percent by weight.

Hard, pore free platinum deposits above 0.0001 inch can be plated with Technic Platinum T. P., according to Technic, Inc., Box 965, Providence, R. I., which says porosity is also negligible for deposits 0.000050-inch thick. Plated from an acid bath at low temperature, the new coating eliminates gassing at the cathode and possibility of gas pitting. Hardness of the deposit is 450 Vickers. Also, Rhenium A plating salts for experimental and high temperature applications such as contacts and sealed relays have been developed by Technic, Inc., Technic reports rhenium has the highest melting point (5732°F) and is second most dense (20.53) of the platable metals.

American Zinc Institute's "Inspection Manual for Hot Dip Galvanized Products" is available to Canadians from the Consolidated Mining and Smelting Company of Canada, Ltd., Box 1030 Place D'Armes, Montreal 1, Canada. Hot dip galvanizing as a protective finish for fixtures and fittings for buildings is discussed in booklet entitled "Hot Dip Galvanizing for Builders" available from the Hot Dip Galvanizers Association, 34 Berkeley Square, London W. 1, England.

Coatings, Organic

Application costs can be cut 30 to 50 percent by a new airless spray vinyl top coating. Tygon AV Series coatings can be built to any reasonable mil thickness with one coat, according to the manufacturer, the Coatings and Lining Division of The U. S. Stoneware Company, Akron, Ohio. The AV Series also has the basic corrosion resistant properties of conventional Tygon top coats, in that they dry to films free of pin

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holes, have longer shelf life and improved chemical resistance.

Preparation and application of vinyl solution resins for coating metal, wood, paper, fiberboard and rubber is discussed in a 24-page bulletin available from B. F. Goodrich Chemical Company, 3135 Euclid Avenue, Cleveland 15, Ohio. Included is a listing of the physical properties of BFG Geon resins suitable for solution coatings.

A new synthetic latex air dry metal primer for automobile bodies and other metal surfaces is reported to possess excellent resistance to water and salt spray by its developer, Chemical Division, Goodyear Tire & Rubber Company, 1144 East Market, Akron 16, Ohio. Called Pliolite Resin Latex 481-X, the resinous, modified styrene-butadiene latex primer can be applied by brush, dip or spray and can be top coated with regular air dry or baked enamel finishes. Excellent adhesion to steel before and after water immersion and good shelf aging of pigmented primers is claimed.

Metals, Exotic

Columbium and columbium alloy sheet, strip, plate, bars and tubes are being produced commercially at Du Pont's new Metals Center at Baltimore, Md. Also the plant is designed to fabricating tantalum, tungsten and molybdenum in the future.

Sintered titanium alloy is now available from the Mechanical Research Division of Cleveite Corporation, 540 East 105th Street, Cleveland 8, Ohio. Designated CPT-64AV, the alloy has tensile strength of 119,000 psi, ductility of 6 percent, elongation and hardness of Rockwell C 23. Strengths to 146,000 psi by heat treatment are claimed.

Metals, Non-Ferrous

Antimony, arsenic, bismuth, cadmium, gold, indium, selenium, silver and tellurium are being produced commercially by American Smelting and Refining Company, 120 Broadway, New York 5, N. Y., to meet the increasing demand for these metals by the electronics industry.

Chromel-AA, a new 80-20 nickel-chromium alloy developed for use as heating elements and mechanical parts in controlled atmosphere furnaces is described in a bulletin available from Hoskins Manufacturing Company, 4445 Lawton Avenue, Detroit 8, Mich.

Non-Metallic

An anchoring compound that mixes with water and expands as it hardens to a rock like consistency within 20 minutes is marketed as Quik-Rok by Preco Chemical Corporation, Westbury, L. I. First used by apartment house builders to secure terrace rail posts in concrete, it also is used to secure light posts in New York City, to anchor heavy posts and machinery and to fill holes created in factory floors by heavy equipment.

Booklet entitled "The Design and Specification of Watertight Concrete" is available from The Master Builders Company, 2490 Lee Boulevard, Cleveland 18, Ohio.

Repairing and resurfacing wooden and concrete industrial floors, concrete, stone, brick, cement and cinder block walls is possible with a new latex modified concrete being marketed as Tuff-patch Hi Test by Shield Chemical Corporation, 251 Grove Avenue, Verona, N. J. Manufacturer claims its tenacious bond to steel makes it ideal as grout for machinery.

Pipe

Zirconium, titanium, columbium, tantalum and other special metal tubing for heat exchangers and piping for the chemical industry will be produced at the Niles, Ohio, plant of Reactive Metals, Inc., which has purchased most of the tube mill facilities from Bridgeport Brass Company. Key personnel of the special metals tube mill have transferred from Bridgeport Brass to Reactive Metals.

X-Tru-Coat pipe coatings, an extruded polyethylene system developed by Republic Steel Corporation in 1958, are offered by both Standard Pipeprotection, Inc.'s new plant at Alton, Ill., and Pipe Line Service Corporation's North Lima, Ohio plant.

Fiberglass reinforced plastic pressure pipe capable of withstanding pressures

to 100 psi at temperatures to 250 F is available from The Ceilcote Company, 4874 Ridge Road, Cleveland 9, Ohio. The pipe, called the Duracor line, is available in 10 and 20-foot lengths of either fiberglass mat or cloth construction. Wall thicknesses range from $\frac{1}{4}$ to $\frac{5}{8}$ inch depending on pressure and temperature requirements. Duracor pipe fittings, including elbows, U-bends, T-joints and other connections, are also available.

Plastics

Threaded elastic polyethylene caps and plugs which protect machine screw threads, pipe threads and tubing during shipping, storage and construction operations are manufactured by S. S. White Industrial Division, 10 East 40th Street, New York 16, N. Y. The elastic protectors fit parts $\frac{1}{4}$ to $2\frac{1}{4}$ inch in diameters ID or OD and have good resistance to acids, alkalis, caustics, petroleum based products and most common solvents. Temperature stability is -95 to 175 F.

A 50-foot self supporting reinforced plastic fume stack exhausts hot corrosive acid fumes from a metal ore refining operation. Built by du Verre, Incorporated, Box 37, Arcade, N. Y., the stack is fiberglass reinforced Hetro resin produced by Durez Plastics Division of Hooker Chemical Corporation, North Tonawanda, N. Y. The 5-foot diameter tank can handle over 70,000 cfm of exhaust fumes and withstand

(Continued on Page 43)

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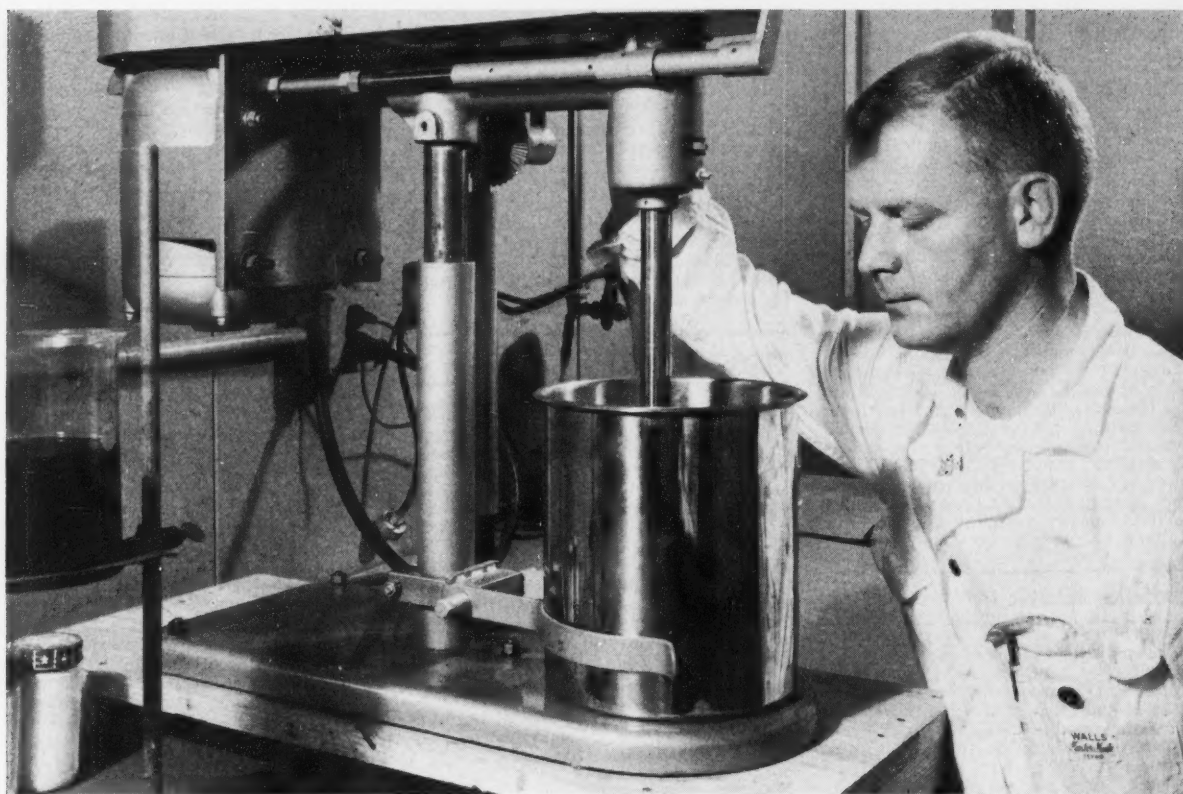


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From TLC Research . . .

TWO NEW PRODUCTS TO RESIST CORROSION

Two more products have joined the growing line-up of new products and processing techniques developed by Tubular Lining Corporation's Research Laboratory. TLC-44 and TLC-77 have been developed to offset salt water disposal and high temperature corrosive conditions.

Here are the product specifications developed by TLC Research:

TLC-44: This is a phenolic resin especially designed to give extreme film flexibility and good resistance to salt water disposal conditions. It is a baked-on multiple film, high-build lining of 6-8 mils. Recommended temperature limitation is 200° F.

TLC-77: This is a special lining designed for elevated temperature conditions found in deep oil and gas wells. TLC-77 is baked on in multiple layers to 4½-5 mils.

It has excellent chemical resistance with good rigid film integrity. Recommended temperature limitations are 350° to 400° F.

New product development is next to quality production in importance at TLC. During the past three years, TLC's Research Department has developed four new lining products for oil field tubing, the "AUTO-TRONICS" and "THERMO-PICKLED" processing methods, and four new testing methods to provide quality linings that do not fail.



NEW . . . For more information about quality control, write for your free copy of *THE STORY OF QUALITY CONTROL*, an eight page description of how Tubular Lining produces a quality product.



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NEW PRODUCTS

(Continued From Page 41)

winds up to 100 mph, du Verre says. While only $\frac{1}{4}$ inch thick at the top, the shell is of tapered construction and rests upon a base flange over 2 inches thick.

Closed-cell elastomeric sponge with good resistance to chemicals, acids and fuels and with high dielectric strength is produced by Industrial Electronic Rubber Company, Solon, Ohio. Possible applications are gaskets, seals and plugs where resistance to grease, oil and commercial gasoline is required, gaskets and seals in presence of perchloroethylene, vibration dampening and insulation. The elastomer compares in hardness to a very soft rubber with a 20 durometer (Shore A) rating but retains the inert characteristics of Fluorel 2141 Elastomer from which it is molded. Sponge comes in sheets up to a half-inch thick and nine-inches square in various pastel colors.

Fluoro elastomeric tubing, hose and press polished sheets resistant to many corrosive chemicals, fuels and lubricants are marketed by U. S. Stoneware Company, Akron 9, Ohio. Die cut and clicked gaskets are also available. Fluoro elastomer used for the tubing is designated Fluran F-5000 and is reported by manufacturer to have operating temperatures to 400 F in continuous service and to 600 F in intermittent service and a brittle point near -40 F. The material can be supplied in hardnesses from 60 to 95 durometer (Shore A scale). At room temperature, tensile strength ranges 2000 to 3000 psi and ultimate elongation from 100 to 400 percent.

New market development prices for the polyethylbenzenes pseudocumene and durenene have been established by Enjay Chemical Company (a division of Humble Oil & Refining Company), 15 West 51st Street, New York 19, N. Y. Pseudocumene (1, 2, 4-trimethylbenzene) in carload drum quantities will sell for 13 cents per pound and in less-than-carload drum quantities for 14½ cents per pound. Durenene (1,2,4,5-tetramethylbenzene) is available in less-than-carload drum quantities at 49 cents per pound.

High temperature resistance fiber (polyamide) will be produced at pilot plant being constructed at Richmond, Va., by E. I. du Pont de Nemours and Company, Inc., Wilmington 98, Del. Main uses of the fiber, designated HT-1, will include electrical insulation, filtration of hot gases, special duty belting and reinforcement of high temperature hose. Completion of the plant is scheduled for late 1962.

Tanks

A 1,000-gallon glass reinforced polyester tank featuring resistance to a wide range of corrosive liquids sells for \$1,000 from Justin Enterprises, Inc., 3755 Edwards Road, Cincinnati 9, Ohio. Bisphenol-A polyester forms inner surface of the tank. Manufacturer claims

tests, copies of which are available on request from manufacturer, have shown that the bisphenol-A polyester is more resistant to prolonged contact with most acids, alkalis and bleaches at elevated temperatures than general purpose or other polyesters. The translucent tank is 10 feet long, 50 inches in diameter and has a wall section of $\frac{1}{2}$ inch; it weighs 300 pounds. Other tanks with capacities of 50 to 10,000 gallons are also available.

Bulk storage of specially denatured alcohol and proprietary solvents is described in a guide available from the Technical Literature Department of U. S. Industrial Chemical Company, 99 Park Avenue, New York 16, N. Y. The guide covers government regulations, suggested tank design including size and location measuring devices and auxiliary equipment such as pumps, pipe and fittings, valves, electrical ground procedures and vents. Illustrated are typical above and below ground storage tanks, gauging well and methods of anchoring underground tanks.

A new liquid processing or storage tank for acids and other corrosives has a chemical resistant seamless plastic inner shell and an aluminum outer shell. Called the Duo-Guard tank, it is 24½ inches long and has an inside diameter of 7 inches and a capacity of 0.5 cubic feet. Bursting strength is 500 psi. The seamless plastic inner tank is available in either Kraylastic B with a working temperature to 185 F or Kraylastic HTHT with a working temperature to 225 F. It is marketed by the Bruner Corporation (a division of Hagan Chemicals & Controls, Inc.), 4767 North 32nd Street, Milwaukee 9, Wis.

Testing

A simplified iron test unit for determining the presence and amount of iron in water is described in a bulletin from Hagan Chemicals & Controls, Inc., Hagan Center, Pittsburgh 30, Pa. Iron test unit KOO82 involves the addition of two reagents to the water sample and comparison with color slides containing nine standards in sealed glass vials representing iron concentrations from zero to 1 ppm.

The Beckman DB Ultraviolet Spectrophotometer for precise qualitative and quantitative analyses of medical, industrial and clinical samples is described in a bulletin published by the Scientific and Process Instruments Division of Beckman Instruments, Inc., Fullerton, Cal. It may be used as a direct reading instrument or with a recorder as a ratio recording system.

A transistorized direct reading conductivity meter which monitors the solids contents of liquids is marketed by Comroe Laboratories, Inc., 5208 South Lake Park Avenue, Chicago 15, Ill. The Autotherm, with its printed and transistorized circuit, weighs only two pounds and operates on an ordinary 9 volt transistor radio battery. It automatically compensates for liquid temperatures from 32 to 212 F.

A four-cubic foot temperature-humidity cabinet for controlled environmental

testing of chemicals, electronic components, paints, plastics, packaging and protective coatings is described in a bulletin available from the Hydson Bay Company, 3070 West Grand Avenue, Chicago 22, Ill. The Environ-Cab provides a temperature range of 0 to 200 F with a control tolerance of plus or minus 2 degrees and a humidity range of 20 to 95 percent with plus or minus 5 percent accuracy.

Miscellaneous

A 68-page booklet entitled "This is Glass," which reviews history of glass, details types of glass and previews future uses of glass and glass-ceramics, is available from Corning Glass Works, Corning, N. Y. Included are a two-page chart giving properties of selected glasses and glass-ceramics and a section on Pyroceram, Corning's new glass-ceramic materials.

An eight-page illustrated engineering guide (Bulletin S-5425) on carbon-graphite materials for mechanical applications is available from National Carbon Company, Division of Union Carbide Corporation, 270 Park Ave., New York 17, N. Y. Includes discussions on high elastic modulus, impermeability, maintained flatness, chemical resistance, thermal conductivity, low friction and wear. Grade recommendations are given for seals and sliding surfaces of various liquids and gases at operating temperatures in the cryogenic range and above 500 F. Properties of National's 18 grades of carbon, graphite and carbon-graphite are given.

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MEN in the NEWS

NACE Member **Frank E. Stetler** has retired from the Wisconsin Telephone Company, 722 North Broadway, Milwaukee, Wis.

Hugh B. Allison and **Joseph S. Hawes** have been elected to the board of directors of Chemical Products Corporation, East Providence, R. I.

Earle B. Barnes has been named general manager and **David L. Rooke** assistant to the vice president of Dow Chemical Company's Texas Division, Freeport, Texas.

C. Edward Bellew has been appointed manager of the Manufacturing Division of Garlock, Inc., Palmyra, N. Y.

Ralph H. Bescher has been named assistant vice president of the Wood Preserving Division of Koppers Company, Inc., Koppers Bldg., Pittsburgh 19, Pa.

James E. Blue has been appointed manager of sales development of Truscon Laboratories, a division of Devoe & Raynolds Company, 1700 Caniff, Detroit 11, Mich.

John B. Hardy, Jr., has been named sales engineer of South Atlantic coastal states by Steele & Associates, Inc., 1405 Dutch Valley Place, N. E., Atlanta, Ga.

A. F. Chouinard, director of research and development, National Cylinder Gas Company, has been elected president of the American Welding Society, 33 West 39th Street, New York 18, N. Y.

NACE Member **Edward A. Roman** has joined the Tape Division of Plymouth Rubber Company, Inc., Canton, Mass., as manager of the Pipe Protection Department.

Robert W. Van Houten, president of Newark College of Engineering, Newark, N. J., has been elected president of the American Society for Engineering Education.

J. H. Westbrook of the General Electric Research Laboratory at Schenectady, N. Y., has been selected by the Electrochemical Society to visit research institutions in the Soviet Union as part of an exchange agreement between the U. S. and the U. S. S. R.

Lorenz H. Wilson has been named director of the Customer Technical Services Department of Sharon Steel Corporation, Sharon, Pa.

The Metallurgical Society of the American Institute of Mining, Metallurgical and Petroleum Engineers has installed three new division chairmen. The Extractive Metallurgy Division is headed by **Richard C. Cole**, executive vice president and general manager of White Pine Copper Company, a subsidiary of Copper Range Company, Boston, Mass. **J. Harry Jackson**, manager of Battelle Memorial Institute's Department of Metallurgy, Columbus, Ohio, is chairman of the Metals Division. **Gerhard Derge**, Jones and Laughlin Professor of Metallurgy at

Carnegie Institute of Technology, Pittsburgh, Pa., heads the Iron and Steel Division.

Robert C. Crowe has been named sales manager of the Cleveland District, Wolverine Tube, Division of Calumet & Hecla, Inc., 17200 Southfield Road, Allen Park, Mich.

Murray A. Wilson, a consulting engineer of Salina, Kan., has been elected president of the National Society of Professional Engineers, 2029 K Street, N. W., Washington 6, D. C.

Paul A. Sieverling has joined Sika Chemical Corporation, Passaic, N. J., as sales manager. **F. M. Ackley** has been appointed technical service engineer and **Alex J. Chidichimo** technical sales engineer.

Shell Development Company, Emeryville, Cal., has appointed four research and development directors at its Emeryville Research Center. **C. W. Smith** is director of exploratory research, **C. R. Nelson** director of industrial chemicals, **J. Anderson** director of synthetic rubber and **R. W. Martin** director of plastics and resins. **S. Z. Perry** has been named manager of the service division, succeeding **J. R. Morrison**, who has become manager of personnel in New York.

Zachary D. Sheldon has joined the Carborundum Company, Niagara Falls, N. Y., as associate director of the research and development division. **Edward J. Finn, Jr.**, has been promoted to manager of the engineering design department, bonded abrasives division.

Murray P. Ellis has been named a regional sales manager for Royston Laboratories, Inc., Pittsburgh 38, Pa.

NACE Member **E. B. Erwin**, with offices in Houston, Texas, has been promoted to district sales manager of Clementina, Ltd., 2277 Jerrold Avenue, San Francisco 24, Cal. Mr. Erwin's district includes Texas, Oklahoma, Arkansas, Louisiana, Mississippi and western Tennessee.

O. B. J. Fraser has retired from International Nickel Company, Inc., 67 Wall Street, New York 5, N. Y.

James H. Gardner has been elected a vice president of National Research Corporation, 70 Memorial Drive, Cambridge 42, Mass. Mr. Gardner will remain general manager of the company's Metals Division.

Charles J. Geyer has been appointed manufacturing and technical director of the Fibers Division of American Viscose Corporation, 1617 Pennsylvania Blvd., Philadelphia 3, Pa.

John R. Ghublikian has been named manager of Process Engineering Section and **Robert G. Hargrove** manager of Project Engineering Section, Badger Manufacturing Company, 363 Third, Cambridge 42, Mass.

(Continued on Page 46)

**REACTS WITH
THE STEEL ...**

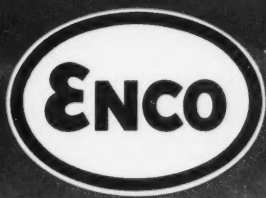
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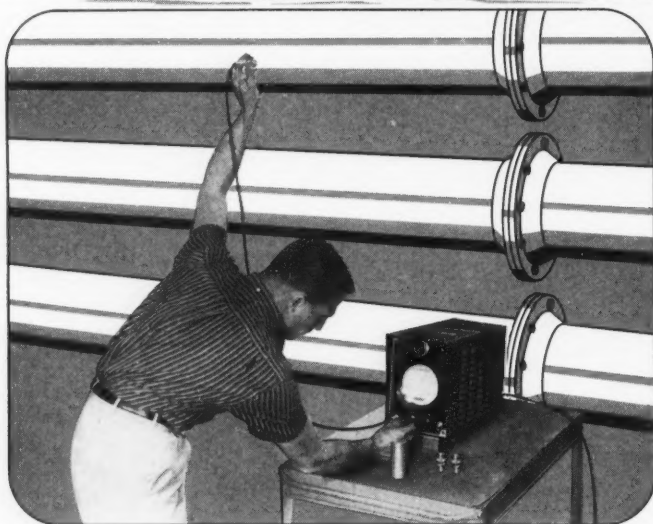
RUST-BAN 190 contains no lead, is non-toxic, non-flammable and comes with curing solution RUST-BAN 195. For complete information, call your Humble salesman or contact Humble Oil & Refining Company, Houston, Texas.



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The Variable Angle Beam Transducer

This new transducer has been specially engineered for angle beam testing with the SONORAY® flaw detector. It incorporates a variable collimator designed to intensify the ultrasonic beam when needed. In addition to standard internal flaw detection, the variable angle beam transducer is also suitable for weld inspection and thickness gaging. The transducer is interchangeable in order to make the frequency fit the job. There are two versions of the variable angle beam transducer presently available: One for continuous water flow and the other with stationary water inside the shoe. The outstanding advantages are:

- Continuously adjustable for all angles, from straight to surface wave.
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- Selection of shoes, flat or curved, to fit the surface of the work piece.

The variable angle beam transducer is further proof of the technical ingenuity and know-how of Branson's Ultrasonic Test Division. The next time you have a testing problem call BRANSON and see how fast BRANSON will find the best solution in the shortest possible time.

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SINCE 1946 — THE RESPECTED NAME IN ULTRASONICS
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MEN in the NEWS

(Continued From Page 44)

Harry Harrison has been named application engineer of the Pump Sales Division of Dorr-Oliver Incorporated, Havemeyer Lane, Stamford, Conn.

Reuben Hitchcock has been named president of Trans-Plastics Corporation, 6700 Morgan Avenue, Cleveland 27, Ohio, a new organization formed to provide process industries with design, development and fabrication of corrosion resistant systems and components made of plastics. **Ted R. Stevenson** is the vice president.

N. P. Hudson has been made sales manager of asphalt protective coatings for Lion Oil Division, El Dorado, Ark., of Monsanto Chemical Company, 800 North Lindberg Blvd., St. Louis 66, Mo. He succeeds **Ray D. Cunningham**, who recently retired.

Joseph C. Kollar has been appointed sales representative of the Cincinnati-Cleveland-Indianapolis sales territory, Pittsburgh Corning Corporation, One Gateway Center, Pittsburgh 22, Pa.

John J. O'Connor has been appointed general manager of production planning and sales service, Stainless and Strip Division, Jones & Laughlin Steel Corporation, 3 Gateway Center, Pittsburgh 30, Pa.

Herman A. Reda has been named District Office sales coordinator of Hagan Chemicals & Controls, Inc., Hagan Center, Pittsburgh 30, Pa.

Thomas T. Spooner and **George H. Dougherty, Jr.**, have been assigned as technical representatives of the South Atlantic and the Mid-Western sales regions, respectively, of Union Carbide Plastics Company, Division of Union Carbide Corporation, 270 Park Avenue, New York 17, N. Y.

Metal & Thermit Corporation, Rahway, N. J., has appointed **Nathaniel L. Remes** as senior research chemist in the organic research and development group. **Earl J. Conway** has been appointed a coatings specialist for the Detroit area.

Nalco Chemical Company, 6216 West 66th Place, Chicago 38, Ill., has appointed three new district managers in the Industrial Division. NACE Member **Orell Collins** will be in charge of the new South Atlantic District with offices in Charlotte, N. C. **R. E. Eddy**, with headquarters in Jacksonville, Fla., will manage the Southeastern District. **P. M. Henry** will head the Mid-South District.

Wolfgang Kreuder has been named manager at Hamburg, Germany, of Deutsche Nalco-Chemie G. m. b. H., a subsidiary of Nalco.

Visco Products Company, Inc., 1020 Holcombe Boulevard, Houston 25, Texas, also a subsidiary of Nalco, has promoted **M. C. Griffin** to manager of the Mid-Continent Region; he succeeds **M. P. Hilbun**, who is the new general manager of Visco. NACE Member **P. H. Mallette** has been named product manager, Corrosion Control Chemicals, at Visco's Houston office, succeeding **J. P. Stanton**, who has been transferred to Nalco's International Division.



NACE NEWS

Western Region Sets 28 Papers For Portland Meeting Oct. 4-6

Twenty-eight papers in eight symposia have been scheduled for Western Region's October 4-6 meeting at Portland. The Benson Hotel will be headquarters. Among the topics covered by the papers are filiform corrosion, thermoplastic piping, corrosion in boiler and steam systems, instruments, heat exchangers, coating design and others. A soil box demonstration of the principles of galvanic corrosion and cathodic protection will be given also.

The schedule follows:

Wednesday, October 4

Corrosion Principles Symposium

Theories and Factors in Filiform Corrosion, W. H. Slabaugh, Oregon State University, Corvallis.

Corrosion of Aqua Ammonia Storage Tanks, L. M. Dvoracek, Union Oil Company.

Soil Box Demonstration, M. C. Miller, Ebasco Services, Inc., New York, N. Y.

New Materials Symposium

Compatibility of Aluminum With Alkaline Building Materials, Robert E. Brooks, Kaiser Aluminum & Chemical Sales, Inc., Spokane, Wash.

New Alloys, Walter Boyd, Battelle Memorial Institute, Columbus, Ohio.

Which Thermoplastic Piping Material for Your Corrosion Problem? Dave Baird, Tube Turns Plastic, Inc., Louisville, Ky.

Tantalum-Titanium Alloys in Mineral Acids, C. R. Bishop, Union Carbide, Niagara Falls, N. Y.

Fresh Water Symposium

Cooling Water System Corrosion, R. A. Loucks, Shell Oil Company, Anacortes, Wash.

Corrosion in Boiler and Steam Systems, J. F. Wilkes, Dearborn Chemical Company, Los Angeles, Cal.

Corrosion in High Purity Water, Ronald D. Weed, General Electric Company, Richland, Wash.

Pure Process Water in Paper Industry, Art Erickson, Weyerhaeuser Timber Company, Longview, Wash.

Thursday, October 5

Cathodic Protection in Practice Symposium

Role of Current Distribution in Cathodic Protection, Robert E. Meredith, Oregon State University, Corvallis.

Cathodic Protection Practices, Harry Brough, Mountain Fuel Supply Company, Salt Lake City, Utah.

Corrosion Testing Instruments, M. C. Miller, Ebasco Services, Inc., New York, N. Y.

Building and Mechanical Equipment Symposium

Metallurgical Analyses of Failures by Corrosion, Harry Honegger, Portland, Ore.

Corrosion in Heat Exchangers, D. B. Gardner, Bell & Gossett Company, Morton Grove, Ill.

Corrosion on Interior of Fuel Tanks, Mark F. Adams, State College of Washington, Pullman.

Corrosion Problems of Aluminum in Building Industry, D. Guy, Alcoa Company, Portland, Ore.

Marine Corrosion and Protective Coating Symposium

Corrosion Testing of Hydrofoil Materials, T. P. May, The International Nickel Company.

History, Properties, Application and Service of Coal Tar Enamels, W. G. Handy, Reilly Tar & Chemical Corporation, Salt Lake City, Utah.

Influence of Coating on Design of Structures, C. G. Munger, Amercoat Corporation, South Gate, Cal.

Friday, October 6

Utilities

A New Look at Materials for Electrical Grounding, C. M. Schilmoller, Los Angeles, and W. K. Abbott, New York, both of International Nickel Company.

Earth Resistivity at Various Depths in Connection With Deep Anode Design, David T. Jones, Pacific Telephone & Telegraph Company, Los Angeles, Cal.

Aluminum Conductors, Bob Dalrymple, Reynolds Aluminum Company, Troutdale, Ore.

Earth Resistivity Measurements for Grounding Grids, Allen L. Kinyon, Bonneville Power Administration, Portland, Ore.

Pulp and Paper Industry

Corrosion Studies on a Groundwood Mill Hydrosulfite Bleaching System, Alan S. Rosenfeld, Crown Zellerbach Corporation, Camas, Wash.

Kraft Mill Corrosion Problems, Robert A. Little, Crown Zellerbach Corporation, Camas, Wash.

Corrosion Problems Encountered in Pulp Bleaching, R. L. Miller, Weyerhaeuser Company, Longview, Wash.

Causes of Corrosion, D. L. Shinn, Crown Zellerbach Corporation, Camas, Wash.

Western Region

San Francisco Bay Area Section was conducted on a tour of U. S. Steel Corporation's Columbia-Geneva Steel Division plant at Pittsburg, Cal., on May 17.

Portland Section heard John W. Caldwell of Bunker Hill Company speak on Cathodic Protection Using Lead-Silver and Zinc Anodes at its May 11 meeting.

Please Note:

The correct dates for the 1961 conference of Western Region are: Wednesday, Thursday and Friday, October 4, 5 and 6.

Previously reported in **CORROSION** (most recently in June, Page 58) were October 3-5.

South Central Region

Permian Basin Section To Hold Tour Sept. 20-22

The 1961 Permian Basin Corrosion Tour of crude oil and gas producing plants in West Texas will be conducted from Odessa, Texas, September 20-22. The biennial event is sponsored by the Permian Basin Section.

The three-day tour includes trips to nine water flood projects to study various water treatment and corrosion control programs of open and closed systems. A wide range of protective coatings, cathodic protection, alloys and chemical inhibitors are employed at these projects.

At producing wells, chemical inhibitors will be explained and discussed. On exhibition will be crude oil handling equipment, including plastic coated storage tanks, flow lines, tubing and sucker rods.

Gasoline plants with their many corrosion problems will be toured and obstacles involved in injecting high pressure sour gas as a secondary recovery method exhibited.

Further tour information and registration forms are available from T. E. Moses, Registrations Chairman, 1961 Corrosion Tour, Box 2749, Odessa, Texas.

Alamo Section saw two films "Corrosion Control With Impressed Current" and "Corrosion Control With Galvanic Anodes" at May 16 meeting. Carl M. Thorn of Southwestern Bell Telephone Company reported on the national convention in Buffalo, N. Y.

Shreveport Section is extending "a measure of financial support" to Centenary College of Louisiana at Shreveport and to the Science Education Council, according to Grady Howell, chairman of the section.

Birmingham Section saw a slide show by W. W. Garrett of Birmingham Industrial Water Works on "Progress of Construction of the Industrial Water Line From Smith Dam to Birmingham" (see June **CORROSION**, Page 32, for article on this steel water line) at May 5 meeting.

(Continued on Page 48)

See Photo Coverage of Appalachian Short Course on Underground Corrosion Pages 52-54

South Central Region

(Continued From Page 47)

Noser, Kleinheksel to Head Symposium on Anodes

Four technical papers have been scheduled as part of the Galvanic Anode Symposium at the 1961 South Central Region Conference to be held October 24-26 at Houston's Shamrock Hilton Hotel.

The four papers are as follows: Magnesium Anodes by Marshall Parker; Zinc Anodes by T. J. Lennox, Jr.; Aluminum Anodes by Ralph Horst, Jr., and a paper on experience records in use of anodes.

The symposium also will include a question and answer period at the conclusion of the session.

Chairman of the symposium is W. P. Noser, one of the founders of NACE, who is senior corrosion engineer with Humble Pipe Line Co., Houston. He has been active in corrosion control work since 1941 and has served as chairman of the NACE technical committees on galvanic anodes.

His co-chairman is Stanley Kleinheksel, NACE corporate member representing his employer, Magnolia Pipe Line Co., Dallas, Texas, where he is corrosion coordinator. He was transferred recently from Plainfield, N.J., where he was engaged in refinery corrosion and metallurgy. He has a PhD in electro-

chemistry from the University of Michigan. Since 1949, his work has been in pipeline corrosion.



Noser



Kleinheksel

The 1963 Annual NACE Conference will be held at Convention Hall, Atlantic City, N. J.

Canadian Region

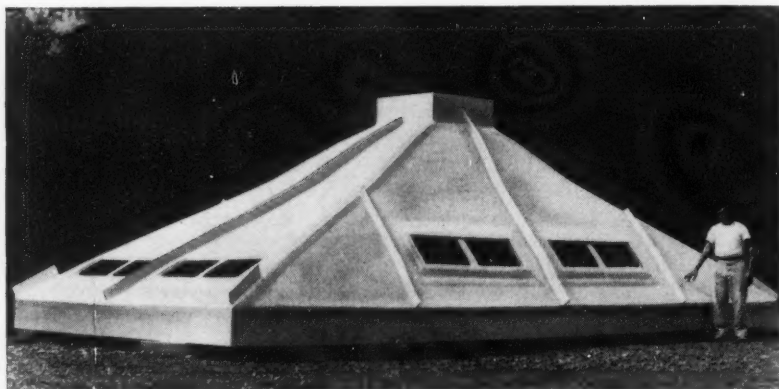
Eastern Division Names Conference Chairmen

Committee chairmen have been named for Canadian Region's 1961 Eastern Division Conference at the King Edward Hotel, Toronto, Ontario, January 17-19, 1962.

General Chairman is H. A. Webster of Corrosion Service Ltd., Toronto. Vice Chairman and Exhibits Chairman is G. K. Dudgeon of B. A. Oil Company

Ltd., Toronto. Program Chairman is W. H. A. Henderson of Electric Reduction Company of Canada Ltd., Toronto. Publicity Chairman is D. Secord, Honeywell Controls Ltd., Toronto. Arrangements Chairman is J. G. Fillo of Bell Telephone Company of Canada, Toronto. Finance Chairman is D. J. Cowan of Corrosion Service Ltd., Toronto. Assistant Exhibits Chairman is G. L. Stone of The Glidden Company Ltd., Toronto.

Edmonton Section heard O. B. Draganiuk, supervisor of corrosion and inspection engineering for Canadian Chemical Company Ltd. speak on Acid Treatment of Power House Boilers and Their Superheaters at May 25 meeting.



ANOTHER BIG CORROSION PROBLEM

Solved by *duVerre*

Size is no problem at du Verre. This huge vent hood is over 20 feet wide and was designed to provide trouble-free corrosive fume removal. Two such units completely filled a large size railroad car.

Over a wide range of corrosive and erosive conditions, du Verre Resin Bonded Fiberglass has proved its superiority over coatings, linings and other traditional "resistant" materials. Completely homogeneous, du Verre fabricated ducts, fittings, hoods and tanks provide uniform protection from flange to flange, inside and out. One-quarter the weight of steel, du Verre fabrications also save you money on freight, handling and erection. Whatever your process needs, equipment can be du Verre fabricated to your specifications. Write today. Ask for Bulletin No. 101 and see how du Verre can eliminate the high cost of corrosion and product contamination.



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TECHNICAL REPORTS

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HIGH PURITY WATER CORROSION

Symposium on Corrosion by High Purity Water by Committee T-3F on High Purity Water which includes:

Introduction to Symposium on Corrosion by High-Purity Water by John F. Eckel.

Corrosion of Structural Materials in High-Purity Water by A. H. Roebuck, C. R. Breder and S. Greenberg.

Corrosion Engineering Problems in High-Purity Water by D. J. DePaul.

The Importance of High-Purity Water Data to Industrial Applications by W. Z. Friend. Per Copy.....\$3.75

Symposium on Corrosion by High Purity Water. Five Contributions to the Work of NACE Technical Committee T-3F on High Purity Water. Pub. 57-22.

Measurement of Corrosion Products in High Temperature, High Pressure Water Systems by A. S. Sugalski and S. I. Williams.

Corrosion of Aluminum-Nickel Type Alloys in High Temperature Aqueous Service by F. H. Krenz.

Corrosion of Aluminum in High Purity Water by R. J. Lobsinger and J. M. Atwood.

The Storage of High Purity Water by Richard R. Dlesk.

Water Conditions for High Pressure Boilers by D. E. Voyles and E. C. Fiss. Per Copy.....\$6.50

Symposium on Corrosion by High Purity Water. Four Contributions to the Work of NACE Tech. Comm. T-3F on High Purity Water. Pub. 58-13. Per Copy.....\$5.50

Corrosion Behavior of Zirconium-Uranium Alloys in High Temperature Water, by W. E. Berry and R. S. Peoples.

Corrosion and Water Purity Control for the Army Package Power Reactor by R. J. Clark and A. Louis Medin.

Removal of Corrosion Products from High Temperature, High Purity Water Systems with an Axial Bed Filter by R. E. Larson and S. L. Williams.

Some Relations Between Deposition and Corrosion Contamination in Low Make-up Systems for Steam Power Plants by E. S. Johnson and H. Kehmna.

Remittances must accompany all orders for literature the aggregate cost of which is less than \$5. Orders of value greater than \$5 will be invoiced if requested. Send orders to National Association of Corrosion Engineers, 1061 M & M Bldg., Houston, Texas. Add 60c per package to the prices given above for Book Post Registry to all addresses outside the United States, Canada and Mexico.

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TYZIN ...a new

zinc-rich primer by Stoneware

Tyzin is a self-curing sacrificial metal primer offering excellent resistance, with or without a top coat, to organic and inorganic solvents, fresh or salt water, oils and greases.

A 2-3 mil film (obtainable in a single application) provides an excellent primed surface for all standard Tygon vinyl top coats or for Tygon Series "E" Epoxy.

Tyzin consists of a modified lacquer base into which is blended metal zinc powder to form a blended solution of better than 57% total solids by weight.

Applied by conventional brush or spray application, Tyzin air dries within 15 minutes to a rain and weather-resistant coating. It can be top-coated with any suitable top coat within a few hours.

With excellent adhesion to properly prepared surfaces, Tyzin proves a low-cost self-sacrificing metal primer for top protection from galvanic attack.

For detailed technical data write
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Stoneware, Akron 9, Ohio.



U. S. STONEWARE
AKRON 9, OHIO

North Central Region



DONALD F. KOENECKE of The Enjay Laboratories, Linden, N. J., who discussed long term lab tests of various coatings at Chicago Section meeting May 16 at the Chicago Engineer's Club, shows slides to Wayne H. Schultz (middle) of Dearborn Chemical Company, Chicago, and Dave Sheldahl of Sinclair Research Laboratories, Harvey, Ill.

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Chicago Section Hears Lab Reports on Coatings

Donald F. Koenecke, The Enjay Laboratories, Linden, N. J. discussed long-term laboratory tests on a variety of coatings before a meeting of Chicago Section May 16 at the Chicago Engineer's Club. Included among the materials tested were various formulations of epoxies and butyls, exposed to environments containing varying percentages of salts, at varying temperatures and with varying voltages of impressed current.

He also demonstrated flame-curing, a process which makes it possible to cure butyl-containing coatings in a few seconds, using fairly high temperatures without unduly heating the substrate. Numerous laboratory data were described and illustrated to make his points. He also described the method of applying coatings by flame spraying in which coating materials are applied through a cone of flame, reach the surface to be coated hot and cure in place.

NACE Publication Director Speaks at Indianapolis

The Indianapolis Corrosion Society, at a meeting May 25 in the Continental Hotel, Indianapolis heard Norman E. Hamner, publication director of the National Association of Corrosion Engineers, Houston, discuss some of the advantages of organizing a section in NACE. Numerous questions were asked Mr. Hamner following his remarks, indicating considerable interest in NACE and its activities. About 15 persons were present.

New officers of the society took over at the meeting. They are: Charles Kleis, president; E. L. Karraker, Shell Oil Co.,

chairman of the Indianapolis Correlating Committee and G. H. Cantwell, Indiana Bell Telephone Co., program committee chairman. Mr. Cantwell is retiring chairman.

Earlier the same day at a meeting of the correlating committee several cases involving underground corrosion were discussed and some mention was made of the scheduled construction of a 20 and 30-inch line to feed underground storage of gas in an old field southeast of Terre Haute. Ralph Willis, Citizens Gas Co., secretary of the committee, presided.

(Continued on Page 56)



TECHNICAL REPORTS

on

CORROSION in UTILITIES

TP-12 Report on Electrical Grounding Practices. Per Copy \$1.50.

T-48 Cell Corrosion on Lead Cable Sheaths. Third Interim Report of Technical Unit Committee T-48 on Corrosion of Cable Sheaths. Compiled by Task Group T-48-1 on Corrosion of Lead and Other Metallic Sheaths. Publication No. 56-9. Per Copy \$1.50.

T-48-2 Cathodic Protection of Cable Sheaths. Fourth Interim Report of Technical Unit Committee T-48 on Corrosion of Cable Sheaths. Compiled by Task Group T-48-2 on Cathodic Protection. Publication 56-13. Per Copy \$4.00.

T-48-3 Tests and Surveys for Lead Sheathed Cables in the Utilities Industry. Second Interim Report of Technical Unit Committee T-48 on Corrosion of Cable Sheaths. Publication 54-6. Per Copy \$5.00.

T-48-4 Pipe-Type Cable Corrosion Protection Practices in the Utilities Industry. First Interim Report of Technical Unit Committee T-48 on Corrosion of Cable Sheaths. Publication 54-3. Per Copy \$4.00.

T-48-6 Stray Current Electrolysis. Fifth Interim Report of Technical Unit Committee T-48 on Corrosion of Cable Sheaths, prepared by Task Group T-48-6 on Stray Current Electrolysis. Publication No. 57-1. Per Copy \$2.50.

T-48 A Concentrator for Coordinated Corrosion Testing—A Contribution to the Work of NACE Tech. Unit Committee T-48 on Corrosion of Cable Sheaths, by Oliver Henderson and Louis Horbath. Pub. 57-26. Per Copy \$1.50.

T-48 Corrosion of Lead Sheath in Man-hole Water—A Contribution to the Work of NACE Tech. Unit Committee T-48 on Corrosion of Cable Sheaths. Pub. 58-6. Per Copy \$1.50.

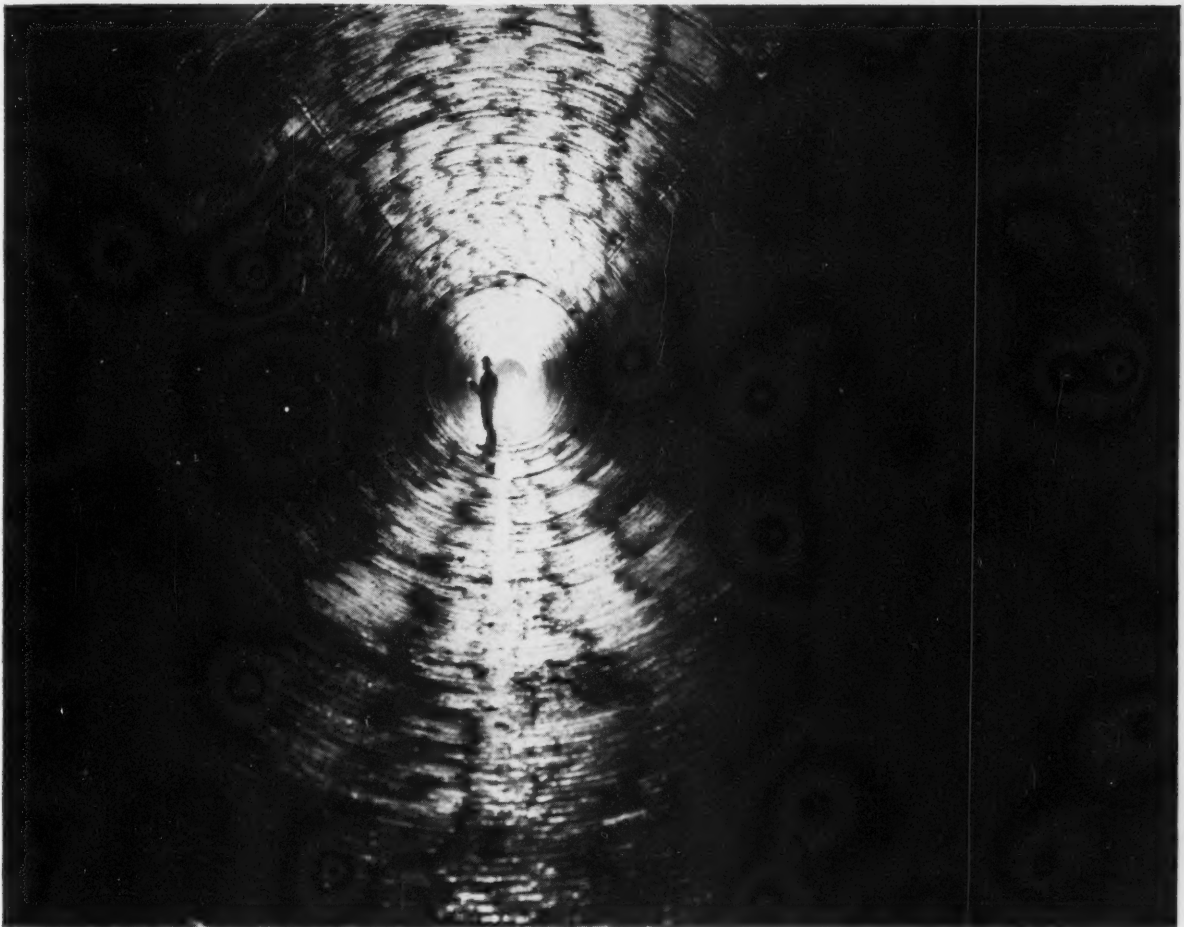
TP-19 Corrosive Effects of Deicing Salts—A Progress Report by Technical Practices Committee 19. Corrosion, January, 1954, issue. Per Copy \$1.50.

T-4F-1 Progress Report of Task Group T-4F-1 on Water Meter Corrosion. Per Copy \$1.50.

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NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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An engineer of Quebec's Hydro-Electric Commission examines the Bitumastic coatings inside the huge Bersimus penstocks.

Bitumastic lining stands up like new in high pressure penstock service

The most critical points in the giant Bersimus River penstocks are the eight 328-foot steel pipe sections, located immediately below the elbows, where water pressure reaches 540 pounds per sq inch. Quebec Hydro-Electric Commission engineers picked Bitumastic coal-tar enamel for the protection of the steel pipe in this tough service.

The coatings applied to this section had to meet very stringent requirements: first, the high moisture content inside the penstocks made it necessary to use a very fast drying primer to protect the coating's bonding action from moisture attack. This requirement was fully met by Bitumastic Jet-Set Primer, the fast-drying primer with a bond estimated to be five times stronger than other primers.

Two coatings of Bitumastic No. 70-B AWWA Enamel were applied to meet the other requirement: these top coatings will have to provide a minimum of five years maintenance free service. A recent over-all inspection of the line indicated that the coatings are still in excellent condition and should last 40 years or more, based on previous experience.

Koppers coal-tar protective coatings are ideally suited for severe corrosion conditions where water resistance and maintenance free service are mandatory. For more information on how Bitumastic coatings can solve your corrosion problems, mail the coupon or write: Koppers Company, Inc.,

Pittsburgh 19, Pa. District Offices: Chicago, Los Angeles, Pittsburgh, New York and Woodward, Ala. In Canada: Koppers Products Ltd., Toronto, Ontario.



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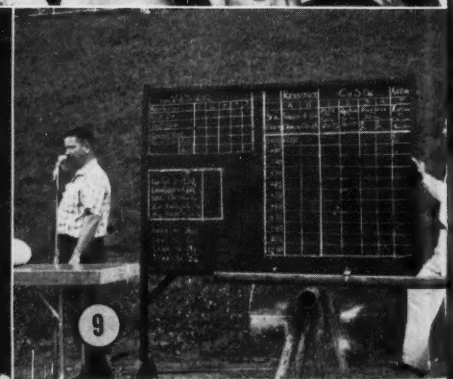
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1961 Sixth Annual

Appalachian Underground Corrosion Short Course



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Photo 1—Part of the bleachers where short course students watched the corrosion testing field demonstration. A 100-foot pipe buried on the campus drill field is used each year during the short course to demonstrate how pipe-to-soil measurements can be made and other test measurements. Photo 2—Registration period before the opening sessions of the course was a time for renewing acquaintances. Photo 3—An interested group watches the demonstration on a corrosion meter at one of the 35 exhibitions on display during the short course. Photo 4—NACE President E. C. Greco of United Gas Corporation, Shreveport, La., gave the opening address. He discussed the economic significance of corrosion control and outlined the activities of NACE in helping industry with corrosion problems. Photo 5—P. H. Miller of Texas Eastern Transmission Corp., Shreveport, La., was the instructor for the basic session on economics of corrosion control. Photo 6—One of the important features of the short course was the Round Table Discussion at which students could ask questions. A panel of seven experts and a moderator was set up to give answers or help on the problems and questions under discussion. Photo 7—Three men from Ohio Fuel Gas Company relax between classes. They are (left to right) William T. James, Jr., of Athens, Ohio, Thomas C. Fisher of Cambridge, Ohio, and W. Wellinghoff of Chillicothe, Ohio. Photo 8—C. A. Erickson, Jr., of Peoples Natural Gas Company gave the mud tub demonstration of the corrosion process and cathodic protection. Photo 9—C. C. Stoneking of Hope Natural Gas Co., at left, described the method being used at the field demonstration to take a soil resistivity survey. At right is F. E. Costanzo of Manufacturers Light and Heat Co., chairman of the field demonstrations. Photo 10—Between classes, small groups such as this continued the discussions or exchanged corrosion experiences. From left to right, they are Charles A. Herdegen of Public Service Electric and Gas, New Brunswick, N.J., J. Edwin Kareha, Public service Electric and Gas, Camden, N.J., David W. Brown of Public Service Electric and Gas, Englewood, N.J., Bob Meihls of Hill Hubbell Co., Middletown, N.J., Joseph C. Sokol of Koppers Company, New York City, and W. J. Skillen of Public Service Electric and Gas Co., Summit, N.J. Photo 11—An outdoor demonstration of a flame spray epoxy coating was given by Minnesota Mining and Manufacturing Company as part of their exhibition at the short course.



Photo 12—Another portion of the bleacher crowd at the field demonstration. For the first time in the short course's six-year history, part of the field demonstration was held indoors because of rain. Photo 13—Waiting for the opening session of the short course is part of the 600 registrants. Photo 14—Tinker and Rasor holiday detectors were demonstrated at the Bob Herrick booth. Photo 15—A thermoelectric generator to power a cathodic protection system was displayed at the Cathodic Protection Service booth. Shown from left to right are Harold C. Boone of Peoples Light and Gas Co., Chicago, Ill., E. C. Paver of Northern Illinois Gas, Bellwood, Ill., and Gordon Doremus of Cathodic Protection Service, Houston, Texas.



Appalachian Short Course Registration Sets Record Again

For the second consecutive year, the Appalachian Underground Corrosion Short Course had a record breaking attendance. Total registration passed the 600 mark to beat last year's attendance of 590.

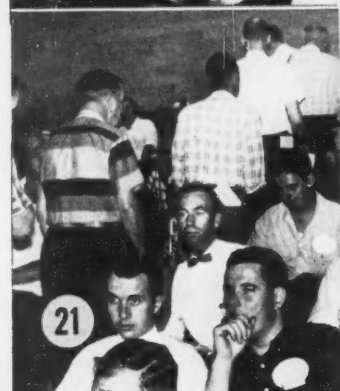
Covering most aspects of underground corrosion, the short course was presented on three levels: basic, intermediate and advanced. Several special topics were added to this year's course including stray current bonds and switches, railroad signal systems, complex structures, bare area protection, well casings, locks and dams, electrolysis defects and tank farms.

General chairman of the short course was D. A. Tefankjian, NACE member with Texas Eastern Transmission Co., Shreveport, La. Program committee chairman was NACE member E. K. Benson of New York Telephone Co., Buffalo, N.Y. He was elected general chairman for the 1962 Seventh Annual Appalachian Short Course.

Dates for the 1962 short course have not been set, pending activities at the University of West Virginia.

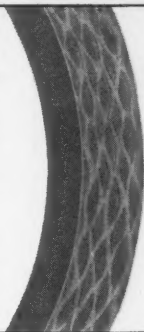
The course is sponsored by West Virginia University and is endorsed by NACE and the American Water Works Association.

Photo 16—Another of the exhibitors at the short course demonstrated the use of special welding techniques for bonding cathodic protection cables to underground structures. Photo 17—Aluminum Company of America had a special display containing a motion picture film. These students are listening to the film's sound track at the Alcoa booth. Photo 18—This between-classes trio is (left to right) I. E. Davis of Ohio Fuel Gas Co., Columbus, Ohio, Fred M. Round of Utilities Service, Niles, Ohio, and R. H. Goodnight of Cook Paint & Varnish Co., Kansas City, Mo. Mr. Goodnight is general chairman for the 1962 NACE Annual Conference to be held in Kansas City next March. Photo 20—The Tapecoat Company was another of the exhibitors at the short course. Here an interested group of students look over the sample pieces of pipe wrapped with plastic tape. Photo 21—Part of the students attending the mud tub demonstration class. Photo 22—Checking their class schedule are (from left to right) James B. Casey of the Tapecoat Co., Ramsey, N.J., Robert H. Teeple of East Ohio Gas Co., Cleveland, Ohio, and Darrel Byerley of Tinker and Rasor Co., San Gabriel, Cal. Photo 23—Five of the many Canadians attending the short course are shown here. All from Bell Telephone Company of Canada, they are (left to right) D. A. Brooks of Montreal, J. A. Clement of Montreal, N. A. McLeod of Montreal, Lauchlan Forsyth of Toronto and Hugh McLean of Montreal.



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3. Operating Temperature Range is: -65° to $+300^{\circ}$ F.
4. Operating Pressure Range Extends to 1200 psi.

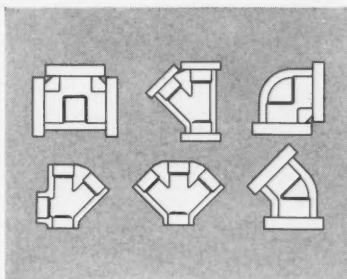
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6. Effectively Stops Electrolytic Action.
7. Fibercast is Unaffected by Extremes of Heat or Cold.

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4. FIBERCAST provides superior resistance to high pressures. Its operating range extends to 1200 psi.
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7. FIBERCAST is unaffected by summer heat or winter cold; has superior resistance to severe atmospheres and weathering.

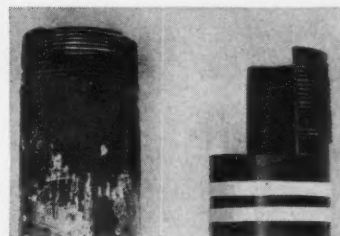


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North Central Region

(Continued From Page 50)

St. Louis Conference Arrangements Completed

Shown in accompanying photographs are some of the conference committee chairmen, symposia chairmen and local section officers who have arranged the 1961 North Central Region Conference to be held October 9-11 at the Chase-Park Plaza Hotel in St. Louis, Mo.

Conference committee chairmen are General Chairman Otto H. Fenner of Monsanto Chemical Co., and his co-chairman Charles W. Ambler, Jr., of American Zinc, Lead & Smelting Co., F. L. Whitney, Jr., of Monsanto Chemical Co., technical program; M. S. Van Devanter of Van Devanter Engineering Co., exhibits; William T. Woodson of St. Louis Metallizing Co., local arrangements; O. W. Siebert of Monsanto Chemical Co., entertainment; Robert Harper of Nooter Corp., printing and publicity; and Mrs. Virginia Siebert, ladies entertainment.

Symposia chairmen for the technical program are C. J. O'Boyle of Metco, Inc., R. G. Kern of Union Electric Co., H. J. Siegel of McDonnell Aircraft Corp., C. L. Griffin of Anheuser-Busch, Inc., W. A. Heideman of Mobil Oil Co., Louis Shaywitz of Transportation Materiel Command, J. F. Revilock of National Carbon Co., S. L. Lopata of Carboline Co., H. O. Nordquist of Joseph T. Ryerson & Sons, Inc., and G. P. Kern of Garlock, Inc.

A complete program for this conference was published on Pages 50 and 51 of the May issue of CORROSION.

Officers of the Greater St. Louis Section, host for the conference, also are among the accompanying photographs: Chairman A. O. Fisher of Monsanto Chemical Co., Vice Chairman M. S. Van Devanter of Van Devanter Engineering Inc., Secretary W. T. Woodson of St. Louis Metallizing Co., and Treasurer Louis Shaywitz of the U. S. Army Transportation Materiel Command.

Southwestern Ohio Section heard J. S. Long of University of Louisville speak on progress and plans for basic research for the paint industry at its May 23 meeting.

New officers for 1961-62 are Chairman Eugene E. Hancey of Armco Steel Corporation, Program Chairman William E. Eckert of Koppers Company, Inc., Treasurer Arthur D. Caster of City of Cincinnati Engineering Department and Secretary Rutson L. Wood of Cincinnati Gas and Electric Company.

New directors are Lewis M. Lederer, Inner-Tank Lining Corporation; Guy H. Cantwell, Indiana Bell Telephone Company; Robert F. Andres, Dayton Power and Light Company; Rex B. Stephenson, Dayton Power and Light Company and Sol M. Gleser, A. M. Kinney, Inc.

Greater St. Louis Section Corrosion Short Course was attended by 65 people from 18 states. The course was held at Washington University June 5-9.



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Northeast Region

Eight Symposia Plus Tours Set for NE Conference

Eight symposia covering topics such as new metals, protective coatings and linings, power generation and transmission, and plastics have been scheduled for the Northeast Region Conference at the Statler Hilton Hotel, New York City, October 30-November 2. Plant tours and an education lecture on anodic and cathodic protection are also scheduled.

The schedule is given below.

Monday, October 30

9-12 a.m.—Power Generation and Transmission. Frank E. Kulman of Consolidated Edison Company, chairman.

2-5 p.m.—Education Lecture: Anodic and Cathodic Protection. George T. Paul of The International Nickel Company, Inc., chairman.

Tuesday, October 31

9-12 a.m.—Saline Water Conversion Units. Dr. W. Sherman Gillman of Office of Saline Water, U. S. Department of Interior, chairman.

Plastics. Byron Zolin of E. I. du Pont de Nemours & Company, chairman.

2-5 p.m.—Plant tours.

Wednesday, November 1

9-12 a.m.—Heat Exchangers. G. Sorell of The M. W. Kellogg Company, chairman.

New Metals. Donald B. Dik of Titanium Metals Corporation of America, chairman.

2-5 p.m.—Protective Coatings. R. P. Devoluy of The Glidden Company, chairman.

Technical Practices Committee meetings.

Thursday, November 2

9-12 a.m.—Protective Linings. Kenneth Tator of Kenneth Tator Associates, chairman.

Evaluation Tools. Harold C. Templeton of Alloy Steel Products Company, chairman.

Genesee Valley Section elected 1961-62 officers at May 11 meeting. Richard Saunders is new chairman, Robert M. White vice chairman and Howard J. Gillen secretary-treasurer. Round-table discussion of corrosion of automobiles by salt during winter was held.

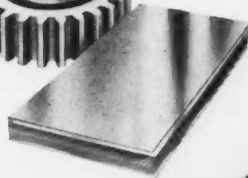
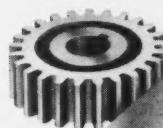
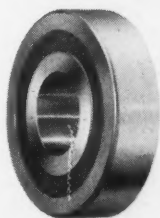
Kanawha Valley Section at May 18 meeting heard Miss Sally Keadle of United Fuel Gas Company, Charleston, West Virginia, speak on Progress in the Natural Gas Industry; she also showed a movie, "The Constant Search," covering the novel compressor driven by a J-57 jet aircraft engine.

Philadelphia Section heard Robert C. DeMarco of Philadelphia Electric Company speak on the Eddystone Electric Generating Station and its underground corrosion problems at May 31 meeting. Fall meeting is set for October 27 at which Robert Jelnick of Syracuse University will present an illustrated lecture on fundamentals of corrosion.

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Southeast Region

SE Region Schedules 17 Papers for Miami

Seventeen papers are scheduled now for the Southeast Region Conference at Miami's Key Biscayne Hotel November 27-December 1. In addition to seven papers announced in June, 1961, CORROSION, ten more papers have been scheduled. These cover such subjects as plastics, sea water conversion processes, coatings and sealants for fuel storage tanks and cathodic protection's effect on corrosion fatigue of steel in sea water.

Authors and papers are listed below.

Coatings and Sealants for Fuel Storage Tanks, Allen L. Alexander, U. S. Naval Research Laboratory.

Mitigative Measures Applied to Underground Lead Sheathed Cables and Associated Hardware, Albert L. Ayers, New Jersey Bell Telephone Company.

The Corrosion Engineer's Choice of Plastics, Otto H. Fenner, Monsanto Chemical Company.

Sea Water Conversion Processes, W. S. Gilliam.

Coating Structural Steel Before Erection, R. J. Koch.

Corrosion of Steel as Influenced by Its Composition and Environment, C. P. Larrabee, U. S. Steel Corporation.

Effect of Cathodic Protection on Corrosion Fatigue of Steel in Sea Water, J. L. Nichols, Dow Metal Products Company.

Austenitic Nickel Cast-Iron in Petroleum Industry, J. F. Mason, Jr., T. P. May and W. K. Abbott, International Nickel Company.

Cathodic Protection—Continuous Measurement and Automatic Control, L. P. Sudrablin, Electro Rust Proofing.

Effect of Cathodic Protection on Corrosion Fatigue of Ship Plate in Salt Water, L. J. Waldron, U. S. Naval Research Laboratory.

Buffalo Paper on Rigid Plastics For Chemical Service Available

A limited number of copies of NACE Buffalo Conference Paper No. 69: Evaluation of Rigid Plastics for Organic Chemicals Service, by L. S. Van Delinder, Union Carbide Chemicals Co. are available for sale, first come first served, \$1 each postpaid. The charge is to defray the cost of handling and mailing of the paper.

The paper, consisting of 26 pages of double-spaced typewriter text, 32 references, 19 tables, and 14 figures, some with as many as four parts, is a thorough summary of presently available knowledge on the subject. A wide variety of test data are included for periods up to 185 days.

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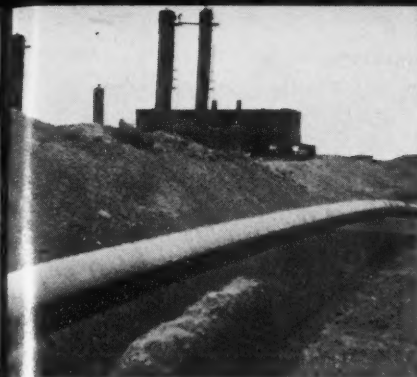
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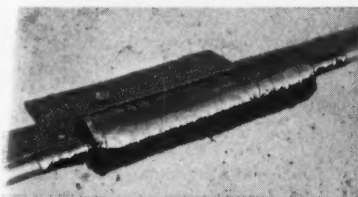
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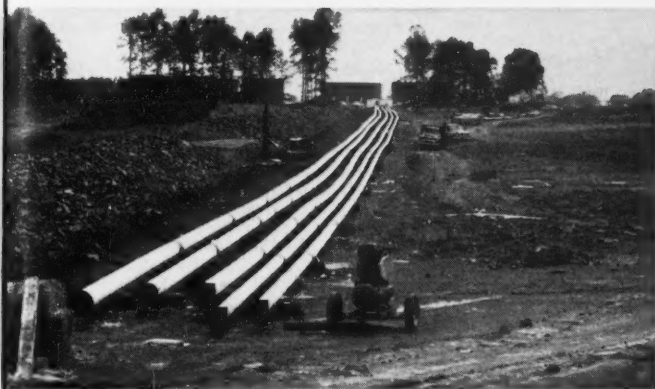


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SECTION CALENDAR

July

- 5 Teche Section
- 18 Chicago Section
- 20 Alamo Section
- 31 New Orleans Section

August

- 2 Teche Section
- 8 Houston Section
- 15 Alamo Section
- 15 Chicago Section
- 28 New Orleans Section

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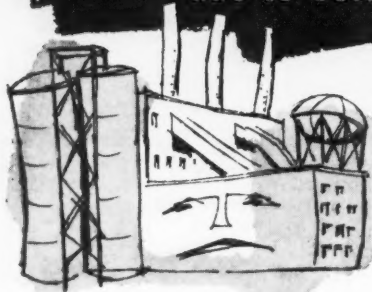
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Technical Articles Scheduled for August

Topic of the Month: A Comparison of the Corrosion Resistance of Basic Oxygen and Open Hearth Steel, by R. C. Talbot, R. N. Hobbs and W. A. Szymanski

A Method for Prevention of Hydrogen Embrittlement of Tantalum in Aqueous Media, by Claude R. Bishop and Milton Stern

Corrosion of Lead Cable Sheath, by K. G. Compton

Use of Pipe-To-Soil Potential in Analyzing Underground Corrosion Problems, by B. Husock

A New Coatings Material for Corrosion Control, by B. H. Mahlman and E. J. Kaatz

A Study of Fouling and Corrosion in Condenser Tubes, by R. A. McAllister, Mitchell Hollier, Donald H. Eastham and Neil A. Dougharty

Use of Differential Thermal Analysis in Exploring Minimum Temperature Limits of Oil-Ash Corrosion, by Norman D. Phillips and Charles L. Wagoner

Microbiological Corrosion in Water-Floods, by J. M. Sharpley

Zinc Filled Inorganic Coatings—A Report of NACE Technical Unit Committee T-6B on Protective Coatings for Resistance to Atmospheric Corrosion, F. P. Helms, Chairman

Recommended Practices for Shop Cleaning and Priming—A Report of NACE Technical Unit Committee T-6D on Industrial Maintenance prepared by Task Group T-6D-4 on Specifications for Shop Cleaning and Priming, L. L. Sline, Chairman

NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1961

Oct. 4-6—Western Region Conference, Benson Hotel, Portland, Oregon.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-26—South Central Region Conference and Exhibition, Shamrock-Hilton Hotel, Houston.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

Nov. 27-Dec. 1—Southeast Region Conference and Florida General Conference Short Course, Key Biscayne Hotel, Miami, Fla.

Nov. 29-30—NACE Board of Directors Meeting, Key Biscayne Hotel, Miami, Fla.

1962

January 17-19—Canadian Region Eastern Division Conference, King Edward Hotel, Toronto, Ontario.

March 18—NACE Board of Directors Meeting, Muehlebach Hotel, Kansas City, Mo.

March 19-23—18th Annual Conference and 1962 Corrosion Show, Kansas City, Missouri, Municipal Auditorium.

March 23—NACE Board of Directors Meeting, Muehlebach Hotel, Kansas City, Mo.

October 1-4—Northeast Region Conference, Hotel Sheraton Ten Eyck, Albany, N. Y.

October 9-11—North Central Region Conference.

October 11-12—Southeast Region Conference, Birmingham, Ala.

October 16-19—South Central Region Conference and Exhibition, Granada Hotel, San Antonio.

1963

March 18-22—19th Annual Conference and 1963 Corrosion Show, Convention Hall, Atlantic City, N. J.

October 14-17—South Central Region Conference, Oklahoma City, Okla.

1964

March 9-13—20th Annual Conference and 1964 Corrosion Show, Sherman Hotel, Chicago, Ill.

October 25-30—North Central Region Conference, Hotel Lemington, Minneapolis, Minn.

1965

March 15-20—21st Annual Conference and 1965 Corrosion Show, Chase-Park Plaza Hotel, St. Louis, Mo.

SHORT COURSES

1961

Sept. 20-22—Permian Basin Corrosion Tour, Odessa, Texas.

Sept. 27-29—8th Annual Central Oklahoma and University of Oklahoma Corrosion Control Short Course, Norman.

Certificates for presentation to past chairmen of regions and sections are available from the Central Office at \$7.50 each.



CORROSION ABSTRACTS

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2.2.5, 2.3.2, 5.3.4

Corrosion Testing of Electrodeposited Coatings. F. L. LaQue. Paper before Am. Electroplaters' Soc., Golden Jubilee Convention, Detroit, June 15-19, 1959. Tech. Proc. Am. Electroplaters' Soc., 46, 141-148 (1959).

Comparative reliability of tests in natural environments and accelerated tests. Role of corrosion tests in research. 37 references.—RML. 19290

2.2.5, 5.4.8, 1.7.2, 2.3.7, 8.9.5

Raft Trials of Underwater Paints. Pts. I, II. J. C. Kingcome. Paint Manuf., 28, No. 12, 375-378, 388 (1958); *ibid.*, 29, No. 1, 5-8, (1959).

Information is given about the site and technique used in carrying out underwater trials of anticorrosive and antifouling paints by Admiralty staff at H. M. Dockyard, Portsmouth. The results of such tests, in conjunction with laboratory evaluations of the coatings, are used to select paints for final trials on ships or marine structures.—RPI. 19254

2.2.3, 2.2.2, 6.2.5, 6.4.2

The Corrosion Behaviour of the Major Architectural and Structural Metals in Canadian Atmospheres. Summary of Two-Year Results. Report by Subcommittee C, Atmospheric Corrosion Testing, E. V. Gibbons, Chairman. 96 pp., Feb. 24, 1959. National Research Council, Associate Committee on Corrosion Research and Prevention, Ottawa, Canada.

Results of examinations of exposed panels after two years are given. Covered are experimental procedures, re-

sults. Metals exposed included aluminum, riveted galvanic couples, ASTM standard steel and standard zinc, low alloy residual, copper bearing and copper-nickel bearing steels; Type 302, 316 and 430 stainless steels; magnesium alloys and rolled zinc. Eight sites are used.

Appendices give detailed weight loss results, weather records, panel identification schedule and membership list of the committee responsible.

Of materials tested stainless steel is reported to have best corrosion resistance, with aluminum next. 19165

2.2.5, 5.4.5

Minimum Paint Film Thickness for Economical Protection of Hot Rolled Steel Against Corrosion. W. Bosch. Official Digest, Federation Paint and Varnish Production Clubs, 31, No. 412, 602-609 (1959).

A report is given on an exposure program on painted steel panels intended to evaluate paint performance in relation to film thickness. Panels were allowed to rust, cleaned by various means, coated with various primer/finish systems (oil, alkyd, phenolic and vinyl) to different film thicknesses and exposed at rural, industrial and marine sites. An interim assessment is presented.—RPI. 19698

2.2.2, 4.2.7, 3.6.6

Tropical Exposure Trials of Bimetallic Couples. (In French.) H. G. Cole. Corrosion et Anticorrosion, 7, No. 1, 3-12, (1959).

Bare and plated steels, and treated and untreated non-ferrous metals and alloys, were exposed for 18 months to tropical jungle (I) and beach (II) conditions. In general, corrosion under II was much more severe than for I. For the coupled metals, anodic corrosion apparently depended on the difference in potential and the degree of "smothering" of the anode by corrosion products. In the choice of couples, account must be taken of the nature of the metals and particularly of the anode. Detailed results and practical recommendations are presented.—MA. 18278

2.2.2, 3.6.6, 6.4.4, 5.9.4, 5.4.5

Report on Marine Atmosphere Exposure of Galvanic Couples Involving Magnesium. A. Gallaccio and I. Cornet. Am. Soc. Testing Materials Special Technical Pubn. No. 255, 1960, 26 pp.

Marine atmosphere exposure study was implemented and completed with objective of acquisition of data and evaluation of performances of various protective systems applied to couples of Mg with Al, Cd (plated on steel), Cu, steel, stainless steel (Type 302), and Zn (plated on steel) to serve as a guide to designers and developers of military equipment. Protective systems Mg included chromate conversion and anodic coatings, and paint systems consisting of chromate primer and alkyd, phenolic and epoxy enamels. Inorganic and organic protective systems for cathode metals included types employed with Mg, except that pretreatments for steel

were phosphate types and pretreatments for Cu and stainless steel were limited to cleaning only. Contact and insulated couples were represented. Appropriate controls included were: uncoupled bare metals; coupled bare assemblies; coupled, pretreated, unpainted assemblies; and several assemblies with paint systems applied to unpainted, coupled metals. Specimens were exposed to Olymic site, approximately one mile south of San Francisco, Calif. Results reported cover an exposure period of 14 months. Map, photos, numerous tables. 24 references.—INCO. 20103

2.2.2

Outdoor Exposure Sites of the National Research Council. E. V. Gibbons. Paper before Chem. Inst. Canada, 42nd Ann. Conf., Halifax, N.S., May 25-27, 1959. Chemistry in Canada, 12, No. 3, 44-48 (1960) March.

Canada now has 7 test sites for study of behavior of metals and protective coatings for metals under outdoor conditions at different locations across country (shown on map). With arrangements completed for reciprocal use of 9 sites of ASTM (including site adjacent to Inco's test facilities at Kure Beach), weather studies can now be conducted at 16 different locations on North American Continent. Climatic conditions at sites vary from severe tropical conditions in Panama Canal

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Zone to low Arctic temperatures of Northwest Territories. Site exposure facilities, specimens used, site calibration (weather records and atmospheric pollution), use of sites, and administration are considered. Metal specimens include Cu-Ni alloy steel, stainless steels, Muntz metal, Monel, electroplated zinc and cadmium on steel, and sprayed zinc and aluminum on steel. Paint systems for steels include 5 types of priming paint and one enamel. A number of different building materials are also being tested. Photos, graphs, 6 references.—INCO. 20191

2.3 Laboratory Methods and Tests

2.3.7, 5.4.3, 2.2.5

A Resume of Procedures for Testing and Evaluating Chemical Resistant Coatings and Linings. R. S. Foster and V. B. Volkening. Dow Chemical Co. Corrosion, 15, No. 10, 85-87, 90 (1959) October.

Methods used by Dow Chemical Company at its Freeport, Texas plant to test coating designed for atmospheric exposures and linings used for inside tanks are described. The need for preliminary screening in the laboratory, on location testing in plant environments or in process streams. Among the considerations important in the selection of linings is the availability of technical service from the material suppliers.

A paper presented under the title "Test Methods for Chemical Resistant Linings and Coatings." Presented at a meeting of NACE Unit Committee T-6A on Organic Coatings and Linings for Re-

sistance to Chemical Corrosion during the NACE South Central Region Conference, New Orleans, October 20, 1958. 18175

2.3.5, 3.2.3

Measurements of Potential/Time on Oxide-Coated Aluminum Surfaces. (In German.) S. Bloeck. Metall, 13, No. 3, 196-198 (1959) March.

It is demonstrated that with potential/time measurements it can be rapidly ascertained whether there is on the surface an oxide or boehmite film present and, whether or not, in a given medium, this film is corrosion-resistant. Furthermore, the measurements provide additional information concerning the degree of corrosive effect of media with different alkalinities. Potential/time measurements also permit the determination of whether the oxide films have been sealed with sodium silicate, or, whether the medium used for corrosion-testing contains any inhibitor. It is pointed out that the method described is not suitable for determining which one of the two kinds of film is present.—ALL. 17618

2.3.5

Measuring Corrosion by the Electrical-Conductance Test Method. Eric Ford. Corrosion Technology, 5, No. 10, 330-331 (1958) Oct.

Decreases in cross-sectional area of specimens due to corrosion are measured by decreases in conductance by a recording potentiometer with constant current flowing through the specimen. Long, thin specimens, (0.01 in. thick) or wires are most suitable. Removal of corrosion products is not necessary and

very low corrosion rates can be measured accurately.—MA. 18756

2.3.9

Use of Radioisotopes in Studying Metal Corrosion and Passivation. (In Russian.) I. V. Krotov. Uspekhi Khim. (Advances in Chemistry), 27, No. 5, 643-667 (1958) May.

Subjects of review are: mechanism of exchange between labeled ions in solution and metal surfaces; isotopic tracer studies of metal-cleaning processes; use of stable and radioactive isotopes in studying certain corrosion processes; tracer uses in corrosion inhibitor mechanism studies; tracer studies on phosphate coatings on metals; and use of autoradiography in studying the properties of metal surfaces. (auth)—NSA. 18816

2.4 Instrumentation

2.4.2, 5.3.4

Electrode Potential Testing. J. H. Bender, Jr., J. W. Dutli and P. D. Edwards. Los Alamos Scientific Lab. Non-destructive Testing, 18, 99-102 (1960) March-April.

A description is presented of the development and some applications of a nondestructive test for ionic surface inclusions in metals, and for pores through an electroplate or cladding. The principle upon which this test method is based involves the electrode potential differences that exists between dissimilar ionic materials when immersed in a suitable electrolyte. The so-called "local action" that takes place in the vicinity of an inclusion or pore produces an electric field with considerable spatial distribution which may be rather easily detected. (auth.)—NSA. 20006

2.4.3

Unconventional Methods. J. Cole. Fulmer Research Inst. Nuclear Eng., 4, 454-455 (1959) Dec.

A method for the assessment of corrosion damage by non-destructive testing is discussed. The deterioration of the specimen with time is measured under a comparatively light load in a four-point loading jig. The deflection under the loading is then measured and the effective thickness of the specimen calculated from the deflection. The inspection of enamel finishes by a high-frequency vacuum probe is discussed. A method of spreading cellulose acetate on a polished surface and subsequently examining the plastic replica of the surface under transmitted light can reveal the early stages of crack formation.—NSA. 20297

2.4.2, 8.4.5

A Sampling Device for Molten-Salt Systems. W. B. McDonald and J. L. Crowley. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-2688, March 21, 1960, 5 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington, D. C.

Design and use of a sampling system which permits the removal of operating-fluid samples from a forced-circulation corrosion-testing loop while the loop is in operation are described. The device is mounted over the one free surface of the fluid and utilizes a dip tube which can be inserted into the liquid and removed without contaminating the inert cover gas of the system. (auth.)—NSA. 20061

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2.4.3, 2.3.6

Non-Destructive Determination of Anodic Film Thickness by Means of Interference Microscopy. (In German.) G. Elssner. *Aluminium*, 35, No. 4, 202-204 (1959) April.

Zeiss interference microscope is described and its use for measuring anodic film thickness, together with the underlying physical principles, are explained. It is demonstrated that this non-destructive method is rapid, accurate and provides information not only on film thickness alone, but also on variations in thickness, surface characteristics and defects. The lower limit of the measuring range is 0.8 μ .—ALL. 17760

2.4.3, 3.8.4, 3.5.8

Can "Exothermic" Electrons be Used for Non-Destructive Testing? (In German.) E. Schmid and K. Lintner. *Schweiz. Arch. angew. Wiss. Techn.*, 24, No. 3, 79-89 (1958).

Cold work causes very low-energy electron emission from ionic crystals on metal surfaces. The emission, originally believed to be due to some exothermic reaction, is related to luminescence phenomena and is now known to be due to the semi-conductive properties of oxide films. The instruments used to detect the emission are described. The phenomenon can be employed in studying the presence, nature, thickness and age of non-metallic surface films, true surface area, surface roughness and surface activity. The emission is stimulated by deformation and in fatigue the strain at which it first appears is much less than in static tests. Rapid assessment of the fatigue limit may be possible by observing the effect. The location of a final fatigue failure was detected after less than half the ultimate life of the specimen in bending-fatigue tests on aluminum strip. 41 references.—MA. 17839

2.4.3, 5.4.5

Assessment of the Porosity of a Protective Anti-Corrosion Coating. (In French.) G. Blet. *Mém. sci. Rev. Mét.*, 56, No. 1, 41-46 (1959).

Conductivity of paint coatings on phosphatized iron, as measured by the porosity meter described, are correlated

with the rates of corrosion in salt-spray. The results are in good agreement. The advantages claimed are that the effectiveness of a coating can be assessed very quickly, and, since it is non-destructive, the porosimeter can be applied to finished assemblies.—MA. 19672

2.4.2, 3.5.4

A Device for the Investigation of the Corrosion and Electrochemical Behavior of Metals under the Action of Ionizing Radiation. (In Russian.) I. L. Rozenfel'd (Rosenfel'd) and Ye. K. Oshe. *Zavodskaya Lab. (Factory Lab.)*, 24, 346-348 (1958). English Translation of *Journal Available from: Instrument Society of America*, 313 Sixth Ave., Pittsburgh 22, Pa.

A description is given of an electrolytic cell which is used to determine the effect of ionizing radiation of the corrosion and electrochemical behavior of metals. The metals being investigated are used as anode and cathode. A measurement of the amperage indicates the course being taken by the corrosion. The investigation shows that irradiation of a cathode sample causes corrosion to increase rapidly, but irradiation does not increase the corrosion of an anode sample. Data from measurements on zirconium-iron are given.—NSA. 18929

2.4.3, 3.2.2, 6.2.5

A Nondestructive Test for Intergranular Corrosion in Stainless Steel. R. C. Robinson. *Am. Soc. Testing Materials, Special Technical Pubn. No. 223* ("Symposium on Nondestructive Tests in the Field of Nuclear Energy"), 112-118, 1958. Available from the Society, 1916 Race Street, Philadelphia 3, Pa.

Variations in resistivity of sound and corroded samples of tubing allow accurate determination of extent of corrosion.—MR. 19022

2.4.2, 2.3.6, 6.4.2

Measurement of Anodic Films on Aluminium. (In German.) W. Illig. *Metalloberfläche*, 13, No. 2, 33-35 (1959) Feb.

An apparatus developed for measuring the thickness of anodic oxide films on aluminum is described. Light incident at a 45° angle is split up into two beams:

one being the result of surface reflection, while the other is due to refraction when the light is entering and leaving the oxide layer and is reflected at the metal-oxide interface. The doublet thus formed may be viewed in a special microscope, and the thickness S of the film can be determined by the formula $S = S' \sqrt{2n^2 - 1}$, where S' is the spacing of the light beams, and n is the index of refraction. Assigning to anodic oxide films a median value of $n = 1.6$, $S \approx 2S'$. Thus, a rapid, non-destructive method of film thickness measurement is presented.—ALL. 17709

2.4.3, 8.8.5

Following the Sintering Processes of Mixture with at Least One Magnetic Component Using the Alternating Field Method. F. Fraunberger and A. Kulb. *Z. Metallkunde*, 50, No. 3, 179-181 (1959) March. (In German.)

Presents simple method to control sintering process in cases where determination of concentration distribution maxima and evaluation of sintering time are sufficient. Instead of usual procedure of measuring Curie temperature for this purpose, more accurate determination of Hopkinson temperature, which is also linear function of concentration, is proposed. Example of application of method to nickel-copper alloys (containing up to 33 copper) is discussed. Graphs.—INCO. 17685

3. CHARACTERISTIC CORROSION PHENOMENA

3.8 Miscellaneous Principles

3.8.1, 3.2.3

Adsorption and Oxide Formation on Aluminium Films. D. D. Eley and P. R. Wilkinson. *Proc. roy. Soc., A*, 254, 327-342 (1960) Feb. 23.

Films deposited at 0° C were stable and with a certain porosity; H_2 and N_2 were not chemisorbed, N_2O and NO reacted giving surface oxide, C_2H_4 gave a slow reversible adsorption and CO gave a slow irreversible adsorption. The kinetics of oxygen uptake were followed in the range 10^{-3} to 10^{-5} mm mercury pressure.—LMB. 20067

3.8.3, 6.3.14

Corrosion Studies. Pt. 8. Passivity of the Sn75-Zn Alloy. (In German.) M. Pražák. *Collection Czechoslov. Chem. Commun.*, 25, 1126-1131 (1960) April.

Investigations were made of the origin of the easy passivation and good corrosion stability of a galvanic alloy of the type 75% Sn-25% Zn. The potentiostatic polarization curve of this heterogeneous alloy was compared with its components in 1 M NaOH. The potential range of the corrosion of tin and zinc in the active state is such that both phases mutually reduce their solution velocity and therefore reduce the critical passivation current of the alloy. The electrochemical behavior of the galvanic alloy does not correspond to the presence of tin and zinc as independent phases and is explained by a passive layer of another composition existing on the independent metals. This layer exhibits in the passive state a low corrosion current which together with the low critical passivation current explains the good corrosion behavior of the alloy. (tr-auth)—NSA. 20017

Materials Deterioration in the Atmosphere

The Mechanism by Which Non-Ferrous Metals Corrode in the Atmosphere by P. M. Aziz and H. P. Godard, Aluminium Laboratories, Ltd., Kingston, Ont.

Mechanisms of Atmospheric Corrosion of Ferrous Metals by C. P. Larrabee, Applied Research Laboratory, U. S. Steel Corp., Monroeville, Pa.

Principles and Procedures Employed in the Design and Interpretation of Atmospheric Corrosion Tests by H. R. Copson, Research Laboratory, The International Nickel Co., Inc., Bayonne, N. J.

Deterioration of Materials in Polluted Atmospheres by J. E. Yocum, Bay Area Pollution Control District, San Francisco, Cal.

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3.8.2

Electrochemical Behavior of the Palladium-Hydrogen System. Pt. II. Thermodynamic Considerations. Gilbert W. Castellon, James P. Hoare and Sigismund Schuldiner. *J. Chem. Physics*, **28**, No. 1, 20-21 (1958).

Thermodynamic relationships between the potentials of the palladium-hydrogen alloys and the known equilibrium properties of the system Pd-H_{gas} , are derived and discussed. A value of 0.048 V. for the potential of the saturated α -alloy derived from the gas-phase data agrees quite well with the observed value of 0.050 V. The spontaneous loss of hydrogen from the electrolytically charged alloy, of composition $\text{H}:\text{Pd} \sim 0.6$, to form an alloy of lower hydrogen-content indicates the presence of an unusual free-energy situation in this system, for which a tentative explanation is offered.—MA. 18851

3.8.4, 6.3.21

A Kinetic Study of the Anodic Dissolution of Germanium. (In Russian.) E. A. Efimov and I. G. Erusalimchik. *J. Phys. Chem. USSR (Zhur. Fiz. Khim.)*, **32**, No. 2, 413-417 (1958).

Anodic dissolution was studied by the polarization curve, potential/time curve, and anode-efficiency methods in 0.1 N hydrochloric acid and sodium hydroxide solution at 10^{-4} - 10^{-5} amp/cm² in N on the (111) face of single-crystal, n- and p-type germanium. Anodic dissolution of n-type germanium at a c.d. of 3×10^{-4} amp/cm² is slower than that of p-type. Anodic efficiency is $\sim 100\%$ calculated on the basis of tetravalent germanium and no gas is evolved. The rate of anodic dissolution of germanium is limited by the rate of diffusion of holes from the bulk of the semi-conductor towards the surface. At higher current density there is a possibility of oxide formation on the germanium surface. A study of the electrochemical behavior of germanium cannot give a true picture of the process without considering the type of its conductivity and the electrical parameters.—MA. 19119

3.8.3, 6.3.8

Anodic Passivation and Depassivation of Lead in Sulphuric Acid. W. Feitknecht. *Z. Elektrochem.*, **62**, No. 6/7, 795-803 (1958).

Stages of the formation of lead sulfate on a lead anode in sulfuric acid solution and the conversion to lead dioxide are followed by electron micrographs and X-ray diffraction. The following reactions occur in the local-cell mechanism: cathodic $2\text{PbO}_2 + 2\text{H}_2\text{SO}_4 + 4\text{H}^+ + 4e \rightarrow 2\text{PbSO}_4 + 4\text{H}_2\text{O}$; anodic $\text{Pb} + 2\text{H}_2\text{O} \rightarrow \alpha\text{-PbO}_2 + 4\text{H}^+ + 4e$. The mechanism of depassivation is discussed. 24 references.—MA. 18818

3.8.2, 8.4.5, 3.5.4

Chemical and Electrochemical Aspects of Corrosion in Nuclear Reactor. (In German.) G. H. Cartledge. Oak Ridge National Lab. Werkstoffe u. Korrosion, **9**, 493-503 (1958) Aug.-Sept. (In French.) *Corrosion et Anticorrosion*, **6**, No. 11, 387-401 (1958) Nov.

Chemical and electro-chemical aspects of the corrosion of metals in reactors are considered. After a survey of the electrochemical processes on the phase boundaries between metal protective films and the solutions under normal conditions, the influences of radioactive radiation on these processes and the corrosion reactions caused by the products of nuclear fission are discussed. Some results ob-

tained at the Oak Ridge National Laboratory are considered. (auth)—NSA. 18857

3.8.3, 6.4.2, 7.2

Electrolytic Passivation of Some Aluminum Alloys. Paul Charlon and Eugene Darmon. *Z. Elektrochem.*, **62**, 794-795 (1958). *Chem. Absts.*, **53**, No. 2, 905 (1959) Jan. 25.

For the preparation of electrolytic valves, aluminum alloys possess advantages over the pure metal. They permit especially the use of neutral electrolytes and alternating current. The time impedance curves obtained on anodic passivation at constant current density shows a linear relation between time and the reciprocal of the current density.—ALL. 18852

3.8.2, 3.8.3, 3.6.8, 6.4.2

Electrochemical Behavior of an Aluminum Electrode in Titration of Aluminum Ion with a Fluoride. (In Russian.) B. N. Kabanov and L. Ya. Polyak. *Zhur. Anal. Khim. (J. Analytical Chem.)*, **13**, 538-544 (1958). *Chem. Absts.*, **53**, No. 6, 4961 (1959) March 25.

Polarization curves in the presence and absence of F^- revealed that F^- caused a shift of 0.3 V toward negative values in the stationary potential of the aluminum electrode. The same shift was observed at the end point when Al^{+++} was titrated with sodium fluoride solution by using an aluminum-nichrome pair of electrodes. The F^- affected only the anodic process, i.e., the dissolution of the aluminum. In the presence of SO_4^{--} and AcO^- , F^- caused a shift in the potential of the aluminum electrode by affecting both the anodic and cathodic processes. Anodic polarization in the absence of Cl^- was accompanied by a shift of potential toward positive values, indicating passivation of aluminum. Thus, Cl^- impeded passivation. Cl^- reduced the hydrogen overvoltage. SO_4^{--} also reduced the overvoltage as well as the current density at which the passivation rise of the curve started. AcO^- reduced the hydrogen overvoltage and in the presence of Cl^- increased the sensitivity of the aluminum electrode to F^- .—ALL. 18795

3.8.4, 3.7.3

Thermal Regeneration of Nickel, Copper and Cobalt After Low-Temperature Oxidation. R. M. Dell. *J. Phys. Chem.*, **62**, No. 9, 1139-1140 (1958).

Dell reviews previous work on, and relevant to, the phenomenon of "thermal regeneration" as displayed by nickel, copper and cobalt after vacuum heat-treatment. Heats of adsorption for oxygen on these metals vary according to their state of aggregation and Dell concludes that the ability of a supported oxide film to reduce its surface free energy by recrystallization accounts for these and other experimental observations. Such recrystallization will be confined to thin orientated oxide films (~ 10 - 20 Å) formed at lower temperatures and the process probably plays a more significant role for nickel than for copper or cobalt. 19 references.—MA. 18141

3.8.4, 6.3.11, 2.3.4

On the Use of Furnaces in the Measurement of the Rate of Oxidation of Platinum and Other Metals (Molybdenum and Tungsten) Forming Volatile Oxides. George C. Fryburg and Helen M. Murphy. *Trans. Met. Soc., Amer. Inst. Mining, Metallurgical Eng.*, **212**, No. 5, 660-661 (1958).

Platinum—and tungsten and molybdenum under certain conditions of high temperature and low pressure—forms an oxide that volatilizes upon formation, so that the surface of the oxidizing metal remains bare, and, therefore, when a furnace is used to determine the oxidation rate the results are too low, since equilibrium is attained between the metal and its gaseous oxide, the measured rate then being determined primarily by the rate of removal of the oxide from the furnace to colder regions. This degrading effect of the hot furnace walls was eliminated by resistance-heating thermocouple-grade platinum ribbons in a cooled tube and collecting the volatilized platinum dioxide, dissolving in 12 normal-hydrochloric acid and analyzing colorimetrically for platinum. At 1200°C under an oxygen pressure of only 150μ mercury the oxidation rate was 100 mg/dm²/day,

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the rate increasing linearly with the pressure up to approximately 250 μ , when the mean free path of the gas molecule is approximately the width of the ribbon used—0.287 cm. Above this pressure the rate starts to fall off from linearity and falls off more and more as the pressure increases.—MA. 18117

3.8.4

Colour of Thin Oxide Films on Metals. A. M. MacSwan. *Proc. Phys. Soc.*, **72**, No. 5, 742-748 (1958).

Origin of the color is considered, and it is suggested that they are due to selective transmission into the medium. This transmission depends on the optical properties of both the oxide film and the underlying metal and on the thickness of the film. The effect is much more pronounced with certain metals. The variation of the composition of the reflected light with film thickness was calculated with a Smith chart.—MA. 18094

3.8.4, 5.9.4, 3.4.8

Effects of Various Anions on Thickness and Active-Oxygen Content of Nickel Oxide Layers formed by Repeated Anodic-Cathodic Cycling; the Specific Effect of Chloride Ion and Its Dependence on the Nature of the Substrate. H. K. Embaby and A. A. Moussa. *J. Chem. Soc.*, **1958**, 4027-4031.

Effect of fluoride, nitrate, perchlorate and chloride on the thickness and active oxygen content of nickel oxide layers formed by anodic-cathodic cycling on nickel and platinum in a nickel salt solution was investigated. At concentrations of 10^{-4} — 10^{-1} Normal, the first three anions had no effect. Chloride at or above 0.02 Normal increased the layer

thickness on the nickel substrate considerably and prevented the formation of free oxygen almost completely. On the platinum substrate there was no effect.—EL. 18029

3.8.2, 5.3.4, 2.3.5

Mechanism of Electrolytic Deposition and Solution of Solid Metals. Pt. II. Processes Occurring During Galvanostatic Switch-On at Silver Electrodes and the Kinetics of Crystal Growth. Heinz Gerischer. *Z. Elektrochem.*, **62**, No. 3, 256-264 (1958).

Course of the time/potential curve at silver electrodes in M-silver perchlorate plus perchloric acid at switch-on is followed oscillographically. The drift of ions through the barrier layer and the deposition or dissolution of silver are two experimentally separable reactions. Total reaction takes place with adsorbed atoms as an intermediate stage. At 25 C these are ~1% of an at. layer. The reaction enthalpy for the formation of adsorbed atoms is ~10.5 kcal/mole. Adsorbed-atom bonds have considerable ionic character. The rate of crystal deposition is three orders of magnitude slower than that of the ion drift. 10 references.—MA. 18761

3.8.2, 6.4.2

Dissolving of Aluminum in Sodium Hydroxide. (In Italian.) Elio Scarano. *Ricerca sci.*, **28**, 1894-1901 (1958). *Chem. Absts.*, **53**, No. 8, 6830 (1959) April 25.

Effect of stirring on the potential of 99% aluminum in deaerated 0.1 N sodium hydroxide was examined. When the sample was horizontal and faced downward, it became 8-10 mv. more cathodic when stirring was stopped; but after one minute the charge reversed and after 30 minutes the potential became constant at a value 50 mv. more anodic than in the stirred solution. When stirring was resumed, there was a transitory anodic jump of 10 mv., after which during 30 minutes, the potential returned to the value it had before stirring was interrupted. When the sample faced upward or was vertical, the potential changes were slight. These observations were attributed to reactions that consumed OH^- faster than H^+ in the vicinity of cathodic impurities, and hence in the absence of agitation lowered the pH. Thus, the cathodes were depolarized and the anodes polarized, and the sample became more cathodic. When the pH had fallen sufficiently, a film of $\text{Al}(\text{OH})_3$ formed. This shifted the potential anodically, since it encroached on the smaller cathodes, whereas its permeability to aluminum ions permitted anodic solution to continue. Corrosion continued in pits adjacent to the largest cathodes, where hydrogen evolution was strong enough to prevent coverage by $\text{Al}(\text{OH})_3$. When stirring was resumed, the pH in the pits rose, further polarizing the cathodes and making the sample still more anodic. Gradually, as the film dissolved, the original potential was restored. On a vertical surface, agitation by hydrogen evolution prevented film formation.—ALL. 18951

3.8.4, 3.5.9, 4.3.2, 3.4.9

High-Temperature Reaction Rates of Several Metals with Hydrogen Chloride and Water Vapor. Milton Farber. *J. Electrochem. Soc.*, **106**, 751-754 (1959) Sept.

An experimental study has been made of the high-temperature reaction rates of various metal filaments with HCl and water vapors and mixtures of the two.

Measurements were made by an electrical method in which the change in resistance of the metal filament was related to the loss in pure metal due to the vapor-metal reaction. Measurable reaction rates were determined for both nickel and iron in a temperature range from 1000 to 1600 K. Specific reaction rate constants and apparent activation energies are presented. The reaction rates of tungsten with water vapor were measured in a temperature range from 1600 to 2000 K. The reaction rates of chromium, Inconel, stainless steel 18-8, and tungsten did not proceed at a measurable rate in either pure HCl or in mixtures of HCl and water. Nickel, copper, Inconel and stainless steel 18-8 did not react appreciably with water vapor at temperatures approaching the melting points. The reaction rates for iron in pure HCl varied from 0.03 to 0.04% area loss per second over the temperature range investigated with an apparent activation energy of 10.5 kcal/mole. The reaction rates of iron in mixtures of HCl and water varied from 0.08 to 0.17% area loss per second in the same temperature range with an apparent activation energy of 8.5 kcal/mole. Reaction rates of nickel in pure HCl varied from 0.005 to 0.03% area loss per second in the temperature range of 1000 to 1600 K with an apparent activation energy of 13 kcal/mole. The reaction rates of nickel in the HCl and water mixtures varied from 0.004 to 0.03% area loss per second in the same temperature range with an apparent activation energy of 22 kcal/mole below 1300 K and 13 kcal/mole above this temperature. Reaction rates of iron in water vapor varied from 0.002 to 0.05% area loss per second from 1120 to 1415 K with an activation energy of 28 kcal/mole. Reaction rates of tungsten in water vapor varied from 0.02 to 0.03 area loss per second from 1700 to 2000 K with an activation energy of 14.5 kcal/mole. 19183

3.8.2, 3.5.9

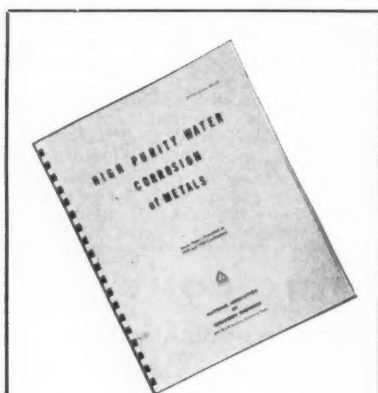
Measurements of Electrochemical Quantities at Temperatures up to 300 C at Equilibrium Pressures. Vilem Pražák. *Werkstoffe u. Korrosion*, **9**, No. 8/9, 524-526 (1958).

An apparatus is described for measuring the change of potential with temperature of HgCl|N-KCl , AgCl|N-KCl and $\text{MgCl|Saturated NaCl}$ electrodes. The latter changes only slowly with temperature, and a high-temperature electrode based on this system is developed. It can be used up to 200 C; its normal potential is +189 mV at 25 C and +179 \pm 2 mV at 200 C.—MA. 18926

3.8.4, 5.9.4

Kinetics of Nitridation of Calcium. M. W. Roberts and F. C. Tompkins. *Proc. Roy. Soc., (A)*, **251**, No. 1266, 369-377 (1959).

Measurements—by a constant-pressure technique—of the kinetics of the reaction of N with sintered, evaporated Ca films at 23-200 C give results consistent with a modified Mott and Cabrera theory of thin-film formation, the rate, dx/dt , being controlled by the mobility of the Ca ions through the nitride lattice, under the influence of the p.d. set up between the metal and chemisorbed N at the nitride surface. $dx/dt \propto p$, the pressure, suggesting a reversible adsorption to form a "surface complex" N_2^- . The activation energy \propto the reciprocal of the thickness of the nitride layer; the critical temperature below which the film grows to its limiting thickness and above which the reaction proceeds unrestricted until



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all the Ca has reacted, is 162 C. The fraction of the total number of metal sites/unit-area-interface from which the Ca ions move into the nitride layer is $\sim 10^{-2}$ – 10^{-4} .—MA. 19433

3.8.4, 3.8.3

Study of the Passivity of Metals in Inhibitor Solutions Using Radioactive Tracers. Pt. II. The Mechanism of Film Growth. O. Kubaschewski and D. M. Brasher. *Trans. Faraday Soc.*, **55**, No. 7, 1200-1206 (1959) July.

A linear relation exists between film thickness and the log. of growth time of very thin oxide films ($< 30 \text{ \AA}$) formed on steel and iron in air and in chromate solution at room temperature. This relation can be explained by postulating that electron transfer through the film is the rate-determining step. 10 references.—MA. 19268

3.8.4, 6.3.21, 3.5.9

Kinetics and Mechanism of High-Temperature Oxidation of Rhenium in the Recrystallized and Cold-Worked States. (In Russian.) V. A. Lavrenko. *Inst. of Metals Ceramics and Special Alloys, Academy of Sciences, Ukrainian S.S.R. Dopovidi Akad. Nauk Ukr. R.S.R.*, No. 11, 1216-1220 (1958).

Temperature dependence equation of the linear oxidation rate constants is calculated for the recrystallized and cold-worked states ($t = 400$ to 725 C). The slowest stages of the process are the kinetically indivisible stages of the electrochemical formation of rhenium oxides and their subsequent vaporization. (auth) —NSA. 19289

3.8.2, 3.8.4, 6.3.11

Exchange Between Metals and Their Ions in Solution. Cecil V. King and Nancy E. McKinney. *Canad. J. Chem.*, **37**, No. 1, 205-212 (1959).

Mechanisms by which a metal can acquire radioactivity when immersed in tracer solution of its ions are discussed, consideration being given to the roles of exchange current, adsorption, local-cell electrolysis, corrosion, and self-diffusion within the metal. Results with silver are presented and the importance of internal diffusion is shown.—MA. 19264

3.8.4, 5.9.4

Faraday's Law and Anodic Dissolution of Metals. Pts. III and IV. (In French.) Michel Froment. *Corrosion et Anticorrosion*, **7**, Nos. 3, 4, 98-109, 134-145 (1959) March, April.

Pt. III. Anodic dissolution of metals by baths containing magnesium perchlorate, ethyl alcohol, perchloric acid, acetic anhydride and sodium chloride aqueous solutions. 14 references.

Pt. IV. Electropolishing of aluminum and aluminum-nickel, and the effect of surface state on the initial valence, are discussed. A conclusion from the study as a whole is that abnormal (from Faraday-law considerations) anodic dissolution is not confined to aluminum and magnesium, as uranium, titanium, lanthanum, cerium, zinc and beryllium in solution can also pass to the unstable-ion state. 19160

3.8.4, 4.3.2, 6.3.21

Kinetics of the Oxidation of Cerium (III) by Concentrated Nitric Acid. R. W. Johnson and D. S. Martin, Jr. *Iowa State College, Ames. J. Inorg. & Nuclear Chem.*, **10**, 94-102 (1959) April.

Oxidation of Ce^{3+} by concentrated nitric acid solutions was shown to be a

reversible reaction. The kinetics for the oxidation at 90 to 110 C in 12 to 16 N nitric acid were studied by a procedure in which volatile oxides of nitrogen were swept away by inert gas. The kinetics were consistent with an electron transfer reaction between Ce^{3+} and NO_3^- , first order in each reactant. An over-all activation energy for the process of 8 kcal was indicated. (auth)—NSA. 19250

3.8.4

Two-Stage Self-Sustained Reaction Between Molybdenum and Potassium Permanganate: Kinetics of the First Stage. R. A. W. Hill and J. N. Welsh. *Trans. Faraday Soc.*, **55**, No. 2, 299-305 (1959).

Chemical kinetics of the first stage of the reaction were obtained between 25 and 110 C by the temperature-profile method. The observed activation energy decreases with temperature, and it is deduced that there is a single reaction path with a succession of energy barriers. At least three such barriers are involved: the first is > 17 , the second approximately 17, and the third is 9 kcal. It is proposed that the first stage of the reaction involves only those MnO_4^- ions suitably placed at the boundaries of the mosaic blocks and reaction occurs between these and molybdenum atoms which have diffused through the permanganate lattice.—MA. 19223

3.8.4, 3.6.5, 6.3.11

Electrode Potentials of the Palladium-Hydrogen System. T. B. Flanagan and F. A. Lewis. *Trans. Faraday Soc.*, **55**, No. 8, 1409-1420 (1959).

Hydrogen was absorbed from 2N sulfuric acid and 2 N hydrochloric acid solutions into cold-drawn and annealed palladium wire. Electrode potentials were measured in the range $[\text{H}]/[\text{Pd}] = 0$ to $[\text{H}]/[\text{Pd}] = 0.69$. Hydrogen content was determined simultaneously by measuring the relative resistance R/R_0 of the palladium wire. Several methods of pre-activating the wire surface were investigated. Agreement was found between

the free energies of solution of hydrogen in palladium calculated from electrode potentials and from gas-phase isotherms. A free-energy difference between electrically charged and gas-phase saturated palladium hydrides cannot be supported.—PMR. 19126

3.8.4, 4.7, 4.6.1

Sodium-Water Reaction Rate Studies. L. Corrsin, H. Steinmetz and B. Marano. *Nuclear Development Corp. of America. U. S. Atomic Energy Commission Publ., NDA-84-19*, May 15, 1959, 32 pp. Available from Office of Technical Services, Washington, D. C.

Experimental techniques have been developed and kinetic experiments have been performed in a sodium-water reaction study undertaken to provide experimental data on the chemical reaction considerations associated with the SDR. Experimental techniques developed for the study of the reaction rate of liquid sodium droplets with liquid water and with steam are described. Results of kinetic experiments on the reaction of sodium with water and deuterium oxide are presented. (auth)—NSA. 19073

3.8.4, 6.4.2

Diffusion of Corrosion Hydrogen in Aluminum Alloys. W. E. Tragert. *J. Electrochem. Soc.*, **106**, No. 10, 903-904 (1959) Oct.

Absorption of hydrogen into aluminum and consequent corrosion is inhibited in 1% nickel alloy by the reduced permeability to hydrogen of the corrosion product film rather than local cathodic discharge of hydrogen.—RML. 18632

3.8.3, 6.3.11

Kinetics of Chlorine Ion Oxidation on Platinum. (In Russian with English Summary.) G. A. Tedoradze. *J. Phys. Chem., USSR (Zhur. Fiz. Khim.)*, **33**, No. 1, 129-136 (1959).

Polarographic and polarization curve methods were used to show that the limiting stage for all processes occurring in the anodic polarization of platinum

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in hydrochloric acid is the discharge of the chlorine ions. A possible mechanism for the oxidation of chlorine is proposed and the retarding effect of oxygen adsorbed on the electrode surface is considered.—PMR. 18572

3.8.4, 3.6.5, 3.6.8

Electrochemical Kinetics of the Anodic Formation of Oxide Films. Pierre Van Rysselberghe and Herman A. Johansen. *J. Electrochem. Soc.*, **106**, No. 4, 355-358 (1959) April.

Potential difference between a metal and a solution across an oxide film is divided into three contributions: (i) the difference at the metal/oxide boundary; (ii) the difference at the oxide/solution boundary; and (iii) the difference across the oxide. (i) and (ii) are both subject to activation overpotential. For one or other of the boundaries the case of linearity of overpotential with log. current (in the range of high currents) or the proportionality of overpotential with current (for small currents) is examined and data for titanium, hafnium and tantalum are analyzed in terms of the theory. Numerical values are obtained for thickness of an interphase, exchange current, p.d., and electrical field at zero current. 11 references.—MA. 18583

3.8.4, 3.6.20

Kinetics of Formation of Anodic Oxide Films in Zirconium. L. Young. *Trans. Faraday Soc.*, **55**, No. 4, 632-642 (1959).

Constant-current kinetics were studied by a spectrophotometric method to measure the thickness. Average field strength in the oxide increases appreciably with increasing oxide thickness

at constant ionic current density. Estimates of activation energy and "half-jump distance" increase with thickness and temperature. Zirconium does not have an overshoot in field strength when the external current is suddenly changed. A model explaining these facts is described. 22 references.—MA. 18652

3.8.3, 6.4.2

Studies of the Structure of Anodic Oxide Films on Aluminum. Pt. I. D. J. Stirland and R. W. Bicknell. *J. Electrochem. Soc.*, **106**, No. 6, 481-485 (1959) June.

Influence of the formation voltage on the structure of nonporous anodic aluminum oxide films has been studied by electron microscope and electron diffraction methods. It has been shown that low formation voltages (< 100 V.) produce an amorphous oxide layer, whereas high formation voltages produce amorphous oxide together with some crystalline γ -alumina. The location of this crystalline oxide within the anodic layer is discussed. (auth)—ALL. 18608

3.8.4, 6.3.15, 6.3.20, 6.3.21, 6.3.9

Oxidation of Titanium, Zirconium and Hafnium. G. R. Wallwork and A. E. Jenkins. *New South Wales Univ. of Tech., Sydney. J. Electrochem. Soc.*, **106**, No. 1, 10-14 (1959) Jan.

A metallographic study of the high-temperature oxidation of titanium, zirconium and hafnium has shown that alterations in the rate equations relating to such processes may be associated with the establishment of oxygen diffusion gradients in the surface layers of the metal. It has been shown that these diffusion gradients in the metal extend in depth during an initial period in which the total rate of oxidation is given by the familiar parabolic equation. Subsequent changes in oxidation behavior have been related to observed alterations in the form of these gradients. (auth)—NSA. 18589

3.8.4

Theory of Formation of High Resistance Anodic Oxide Films. L. Young. *Canadian J. Chem.*, **37**, No. 1 276-285 (1959) Jan.

Various models are considered for the growth of anodic oxide films (metal ions mobile). In general, a transition is expected, as the thickness of the film is increased from control by the metal/oxide interface (Cabrera and Mott) with very thin films to control by the movement of ions through the body of the film (Verwey), with the concentration of mobile ions taking up the value (p , say) which gives electroneutrality. The field strength only varies with thickness in the transition region of thickness. Dewald's theory is the special case of p zero, which gives a field increasing continuously to infinite thickness. The high field production of Frenkel defects (with the vacant cation sites immobile and the interstitial ions mobile) as postulated by Bean, Fisher and Vermilyea and a slight mobility of oxygen ions are two processes which would allow p to vary with the field strength, and which would, therefore, give rise to "overshoot" in the transients in the field strength which occur when the applied current is suddenly changed. However, if the field strength is sufficiently great to produce Frenkel defects it would be expected to be sufficiently great to cause the vacancies to be mobile. This case is considered.

Finally, it is noted that in an amorphous oxide it is difficult to maintain a distinction between lattice and interstitial ions, and, in fact, a range of site energies and jump distances would be expected. Some of the observed features (including "overshoot", and Tafel slope anomalies) of the kinetics for tantalum may, therefore, be due simply to the fact that the oxide is amorphous. (auth)—ALL. 18711

3.8.4, 6.3.21

Kinetics of the Oxidation of Antimony. A. J. Rosenberg, A. A. Menna and T. P. Turnbull. *J. Electrochem. Soc.*, **107**, 196-199 (1960) March.

Reaction rate between 265 and 385°C is initially parabolic, degenerating to a time-independent rate controlled by the diffusion of gaseous $(\text{Sb}_2\text{O}_3)_2$ from the surface. A layer of Sb_2O_3 forms on the surface and supports a further growth of Sb_2O_3 . 10 references.—RML. 20030

3.8.3, 3.8.2, 6.2.2, 4.3.2, 3.2.2

Stability of Heterogeneous Surface Structures During the Corrosion of Passivable Metals. (In German.) U. Franck. *Werkstoffe u. Korrosion*, **11**, No. 7, 401-410 (1960) July.

Question of the stability of surface structures of corroding metals is examined on the basis of the thermodynamics of irreversible processes, and it is shown that, in the presence of several independent variables of state, even heterogeneous structures such as those encountered with pitting corrosion may be stable. This is demonstrated in the Second Part by means of a practical example, quantitatively calculated by an analogue computer, concerning the corrosion of iron in sulfuric acid. By way of application, a theory is developed in the Third Part concerning the pitting corrosion of iron in sulfuric acid containing halide ions; this theory is largely based on the nonmonotonic alignment of the current potential curve of the passivable metals. 20039


3.8.4, 6.3.16, 2.3.6

Kinetics of Strontium Oxide on Tungsten. J. A. Cape and E. A. Coomes. *Univ. of Notre Dame. J. Chem. Phys.*, **32**, 210-214 (1960) Jan.

Field electron microscopy was used to study the kinetics of strontium oxide on tungsten in the range 900 to 2200 K. Reactions take place on the [111] and [100] which have high concentrations of four nearest neighbor surface atoms. Below 1150 K there is some evidence for the dissociation of SrO and migration of Sr to the [110] edges where clusters of crystallites are formed. Two modifications of field emission patterns associated with the (Sr-O-W) complex within 1150—1550 K are tentatively identified with the basic and normal tungstate; apparently the first goes over to the second with an activation energy of ~ 60 kcal as determined from desorption data. An unusually sharp decrease in Fowler-Nordheim work function occurs during this reaction. In the later stages of desorption above 1550 K patterns identifiable with an oxygen-tungsten surface are obtained. Desorption at this stage takes place with an activation energy of ~ 140 kcal. (auth)—NSA. 20261

3.8.4

Investigation of the Role of Gaseous Diffusion in the Oxidation of a Metal Forming a Volatile Oxide. J. L. Modis-



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ette and D. R. Schryer. National Aeronautics and Space Administration, Technical Note D-222, March, 1960, pp. 1-13.

A rate equation expresses oxidation rate as a function of temperature, gas properties, flow conditions and activation energy at temperature at which the oxide is gaseous for tungsten, tantalum, molybdenum, niobium, zirconium and titanium. Oxidizing gas mixtures are flowed over specimen at 1 to 7 t/sec between 1900 and 3000°R. 7 references.—RML. 19838

3.8.3, 6.2.5, 2.3.5

Electro-Chemical Measurements on the Passivation of a Steel Containing 18% Chromium and 8% Nickel. (In German.) A. Rahmel and W. Schwenk. Arch. Eisenhüttenw., 31, 189-193 (1960) March.

Current density-time relations during the potentiostatic passivation of an 18% Cr—8% Ni steel in sulfuric acid and a sodium sulfate solution. Current density-time-relations at potentials vary from +150 to +1000 mV_N within the vicinity of passivity. Interpretation of the results with respect to the kinetics of the formation of passivated layers. Discussion of the formation of very thin oxide layers.—RML. 20008

3.8.2, 3.6.6, 6.3.17

Corrosion of Construction Materials, Bonding Materials, and Uranium—An Electrochemical Investigation. R. B. Hoxeng. Chicago Univ. U. S. Atomic Energy Commission Pubn., CT-3023, May 2, 1945 (Declassified Jan. 28, 1960), 70 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington, D. C.

A study was made of the effect of

solution composition on the galvanic corrosion of the (72S)2S Al couple. Equipment is described which was used to obtain electrochemical information. Results are presented graphically. Preliminary studies of bonding materials showed that Sn—Zn solders of certain compositions could be heat treated so as to retain enough Zn in solid solution to have a solution potential cathodic to Al. Solution potentials between different portions of a pitted U specimen may be as high as one volt and are probably an important factor in the corrosion of uranium.—NSA. 20009

3.8.4, 3.4.9

Metal-Water Reactions. Pt. 6 Analytical Formulations for the Reaction Rate. Leo F. Epstein. General Electric Co. U. S. Atomic Energy Commission Pubn., GEAP-3272, September 30, 1959, 52 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington, D. C.

Various types of equations relating the extent of reaction between a metal and water vapor are reviewed and discussed. The origin of the most common relation employed, the so-called parabolic rate law, is shown to be the diffusion process through an oxide film on the surface. It is shown that such a law cannot apply to the initial stages of the reaction process, before a coherent film has had a chance to form. For these conditions it is postulated that the rate determining process is the transport of H₂O molecules through the vapor phase. An exact formulation combining these two concepts, and an empirical representation are presented. Other rate laws, such as the cubic, logarithmic, and linear forms are considered. The dependence of the reaction rate on pres-

sure and temperature is analyzed, and it is shown that neither is sufficiently well known to permit more than approximate computation. The effect of geometry on the rate law is discussed by considering plane, cylindrical, and spherical configurations. Equations for determining the rate and extent of reaction when the temperature varies with time are considered. (auth)—NSA. 20131

3.8.2

Electrochemical Corrosion of Metals. (In Russian.) A. I. Krasilschikov. Khimicheskaya Nauka i Promyshlennost', 3, No. 4, 476-483 (1958).

Theoretical. In most cases, if the metal is not in the passive state the total corrosion rate is determined by the rate of cathodic processes. During the study of O reduction on Ag, Au, or Hg cathodes the first to be reduced were the mol, and not atoms of O, forming in the first place mol. of H₂O₂ which either decomposed or were reduced further. The kinetics of the processes were basically the same in acid and alkaline solution, the only difference being in the limiting stages. On Pt and Pd, however, O from the solution combines with H adsorbed or discharged on the cathode. Starting with Tamm's hypothesis [Physikal. Z. Soviet-union, 1, 733 (1932)] of the two-dimensional electron cloud, Krasilschikov studied the influence of the quantity of adsorbed gas on passivity, and derived a relationship according to which the change in the energy of adsorption on transition from a free surface to one completely covered by the gas is 3.8×10^{-10} erg/electron. Hence, even a small quantity of adsorbed O retards considerably the

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rate of metal dissolution. Local or pitting corrosion is caused by a thermal movement of mol. at a point, changing the potential at that point. This is enhanced by differential aeration effects. Anodic dissolution of metal or H can proceed through direct transfer of ions into solution or through chemical interaction of the metal with mol. in solution forming intermediate products such as OH^- . 81 references.—MA. 20031

3.8.2, 6.3.21

On the Mechanism of Anodic Dissolution of Germanium in Alkaline Solution. Fritz Beck and Heinz Gerischer. *Z. Elektrochem.* **63**, No. 4, 500-510 (1959).

Electrodes were made from germanium single crystals—p-type doped with antimony to 0.07, 0.3, 1.0, 11 and 27% cm and n-type germanium doped to 0.08, 1.5, 6.6 and 142 cm—and used to obtain stationary current/voltage curves galvanostatically and potentiostatically. The rate-determining step of the dissolution is exponentially dependent on the potential, and for OH^- ions and defect electrons is a first-order reaction. The dissolution mechanism proposed by Turner (*J. Electrochem. Soc.*, **103**, 252, 1956) is rendered more precise. 20 references.—MA. 20166

3.8.2, 3.4.6

Effect of Oxygen on the Work-Function of Barium. P. A. Anderson and A. L. Hunt. *Phys. Rev.*, **115**, No. 3, 550-552 (1959).

Determinations were made by the contact-p.d. method, referred to a standard surface. The work-function of clean barium decreased by 0.32 eV on sudden exposure to a large dose of oxygen, remained constant at this value for 5 days, and then slowly drifted back to the original clean-barium value. No change was observed in the latter on slow admittance of oxygen, even after the addition of an equivalence of 100 monolayers of the oxide.—MA. 20276

3.8.2, 4.7

Thermodynamic Evaluation of Materials in Contact with Fluoride Fuels. R. C. Crooks, D. E. Sparrow, J. J. Ward and R. B. Filbert, Jr. Battelle Memorial Inst. U. S. Atomic Energy Commission Pubn., BMI-936, August 5, 1954 (Declassified Sept. 9, 1959), 19 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion tendencies of 44 metallic elements, nonmetallic elements, and metal oxides, borides, and carbides were evaluated by means of thermodynamic methods. Standard free energies of reaction and equilibrium constants are compiled for 1200 and 1500 F. The effect of activity coefficients on the thermodynamic corrosion tendency is discussed, together with the effect of complexing actions that alter the effective concentrations of the corrosion reactants and products. Emphasis is placed on the fact that a material that chemically has a strong tendency toward corrosion may be corrosion resistant in practice because of physical factors that protect the material. (auth) —NSA. 20243

3.8.4, 6.3.20, 8.4.5

Effect of a Thermal Gradient of the Hydrogen Distribution in a Metal. Application to the Sodium Reactor Experiment. G. G. Libowitz. Atomic International Div., North American Aviation, Inc. U. S. Atomic Energy Commission

Pubn., NAA-SR-4194, December 1, 1959, 20 pp. Available from Office of Technical Services, Washington, D. C.

Distribution of a gas dissolved in a metal under a thermal gradient and constant pressure is considered. Expressions for the concentration gradient and the over-all concentration necessary for compound formation are derived. The derived equations are applied to the problem of the distribution of hydrogen in the zirconium cans of the Sodium Reactor Experiment. It is shown that if the over-all hydrogen concentration exceeds 4×10^{-4} atoms of hydrogen per atom of zirconium, δ -phase zirconium hydride will form at the cooler part of the zirconium cans. (auth)—NSA. 19891

3.8.4, 6.3.6, 4.4.8

Kinetics of the Dissolution of Copper in Oxygen-Containing Solutions of Various Chelating Agents. J. Halpern, H. Milants and D. R. Wiles. *J. Electrochem. Soc.*, **106**, 647-650 (1959) Aug.

Kinetics of the dissolution of copper in stirred oxygen-containing solutions of ethylenediamine and several amino acids have been examined and compared with those found earlier for ammonia. In each case, provided that the partial pressure of oxygen exceeds a critical value (below which its transport to the copper surface may be rate limiting), the rate was found to be zero-order in oxygen and first-order in the complexing agent. The rate-determining process is believed to be the chemical attack of the oxygen-covered copper surface by the complexing agent. At 25°, the rate constants for dissolution by ethylenediamine, glycinate, alpha-alaninate, and beta-alaninate were found to be 2.3×10^3 , 49, 59, and 11 mg Cu cm⁻² hr⁻¹ M⁻¹. These follow the same order as the stability constants of the corresponding cupric complexes. In each system there is also another reaction path, independent of the first, which involves the protonated species, i.e., the ethylenediaminium ion and the neutral amino acids. The corresponding rate constants are 5.1×10^3 , 31, 36, and 3.5 mg Cu cm⁻² hr⁻¹ M⁻¹. 19853

3.8.4, 6.3.16

Adsorption of Oxygen on Tungsten. Joseph Eisinger. *J. Chem. Physics*, **30**, No. 2, 412-416 (1959).

From a room-temperature study of the adsorption of oxygen on single-crystal tungsten ribbon it was found that the maximum coverage is 12.3×10^{14} atoms/cm², and the initial sticking probability is ~ 0.24 . These results are discussed in terms of an idealized atom model for the tungsten surface. A study was made on the rate of oxide formation at the surface of the hot tungsten filament. 13 references.—MA. 20069

3.8.4, 3.7.4, 6.2.2

Diffusion in Metals. Progress Report and Publication List. David Lazarus. Illinois University. U. S. Atomic Energy Commission Pubn., AECU-4186, June 1, 1959, 18 pp. Available from Office of Technical Services, Washington, D. C.

Iron oxidation studies are reported for single crystal whiskers; correlation of oxidation nuclei with dislocation structure is difficult and study is being directed toward oxidation while the crystal is under stress. Oxidation at grain boundaries was also studied as well as hydrogen reduction of the oxide. —NSA. 19832

3.8.4

Oxidation of Metals: The General Oxidation Equation. (In English.) Tor Hurlen. *Acta Chem. Scand.*, **13**, No. 4, 695-704 (1959).

From the absolute reaction-rate theory, a general oxidation equation is stated for transport of metal and oxide vacancies and interstitials in ideal solution in the oxide film, under the action of chemical and electrical potential fields. This manipulated to give inverse log, cubic, and parabolic oxidation equations, and the assumptions made for this purpose lead to the association of a range of film thicknesses with each type of equation, such that a complete oxidation/time observation embraces an initial period of inverse log oxidation and changes smoothly to a period of cubic and general oxidation and finally parabolic oxidation. The regions of validity of each rate equation are calculated for the case of Ti oxidized to TiO_2 , and presented graphically in terms of film thickness at the oxidation temp. The conditions for cubic oxidation do not require the assumption of a p-type oxide. 22 references.—MA. 19815

3.8.3, 3.6.8, 6.3.2

Anodic Oxidation of Cadmium. Pt. 2. Electrical Properties of the Film. Phillis E. Lake and E. J. Casey. *J. Electrochem. Soc.*, **106**, No. 11, 913-919 (1959).

Cd anodes were examined under anodic oxidation in 0.72-14.4 M-KOH and 0.72-7.2 M- K_2CO_3 by measuring overpotential decay after current interruption. After passivation, while O is being evolved, the potential decays log, and before passivation, while Cd is being oxidized, the decay is exponential in two steps. The capacitances of both active and passive Cd were measured by potential-decay curves and by superimposed a.c. methods. Values obtained by decay methods before passivation are two orders larger than a.c. values. It is proposed that, in addition to the Helmholtz double layer, an inner layer is present in which OH^- is reduced to O^{2-} . When the field strength across the inner layer becomes high enough, $\text{OH}^- \rightarrow \text{OH}^\cdot$ and O^{2-} is evolved. The rates of OH^- and CO_3^{2-} are discussed. 17 references.—MA. 19803

3.8.4, 6.3.6, 2.3.6

Length Distribution of Whiskers Formed During the Oxidation of Copper. W. Jaenicke and L. Albert. *Naturwiss.*, **46**, No. 16, 491 (1959).

An electron-microscope study was made of the growth of oxide whiskers formed on sheet specimens of electrolytic copper exposed to O (150 mm Hg pressure) at 300-500 C. The whisker lengths were measured as a function of oxidation time from stereographic photographs of C replicas of the metal surface. The length distribution was found to be of the form: $dN(d\lambda, N=k \cdot \exp(-k\lambda))$, where λ is whisker length, N the number of whiskers. The constant k increased as $t^{1/2}$, where t is the oxidation time. It is concluded that: rate of nucleation \propto the rate-of-defects development in the Cu_2O -CuO layer due to mechanical distortion; growth is by surface diffusion of Cu and O to give $\lambda = at^{1/2}$, where $a = 2 \times 10^{-4}$ cm/min^{1/2} at 500 C; needle growth is stopped by a statistical inhibition process which proceeds independently of whisker length. Inhibition is due to impurities.—MA. 20114

3.8.2, 6.3.21

Electrode Processes on Germanium in Sulfuric Acid Solutions in the Presence of an Oxidizer. E. N. Paleolog, A. Z. Fedotova and N. D. Tomashov. Doklady Akad. Nauk, SSSR, 129, No. 3, 623-626 (1959). Translation available from Morris D. Friedman, Inc., P. O. Box 35, New Newton 65, Massachusetts. (Order MDF P-140).

Kinetics of the electrode processes on *n* and *p*-type germanium single crystals in an H_2SO_4 (pH 1.0) solution with admixtures of H_2O_2 and Fe^{3+} was investigated during a study of the self-dissolution mechanism of semiconductors. All experiments were conducted at a temperature of $25 \pm 0.1^\circ$, in the dark. Results indicate that the type of germanium conductivity exerts a large effect on the rate of the electrode processes occurring thereon. The cathodic reactions reducing the Fe^{3+} and H_2O_2 and the hydrogen-ion discharge were strongly accelerated for *p*-type germanium, while the anodic dissolution process for *n*-type germanium was made difficult. The cathodic reactions on *n*-type germanium apparently proceed with the predominant participation of free electrons. The strong deceleration of the cathodic process for *p*-type germanium can be related to (1) the insufficiency of free electrons under the condition of their predominant participation in the reactions reducing the Fe^{3+} and the H_2O_2 , and (2) the additional ohmic potential drop in the surface of the *p*-type germanium which is impoverished of holes. In the latter case the valence electrons are the fundamental participants in the reduction reactions. The rise in the anodic saturation current density for *n*-type germanium in the presence of Fe^{3+} can be related to either (1) the specific adsorption of these ions on the germanium surface and the consequent increase in the recombination rate near the germanium surface layer, or (2) the simultaneous occurrence of the reaction reducing the Fe^{3+} ions on the anode, which leads to a growth of the hole concentration. 6 references.—TT. 19995

3.8.2, 6.3.11

Potential of the Platinum Electrode in Heterogeneous Gas-Aqueous Solution Systems. G. Bombara. Metallurgia Italiana, 51, No. 3, 101-108 (1959).

A plate 0.5×0.5 cm and spiral wires 8×0.7 mm in diameter were used after cathodic and anodic treatments and immersion in chromic or acetic acid-hydrogen peroxide solution, and also after oxidation with $\text{Ce}(\text{SO}_4)_2$. The solutions used were of sodium chloride; $\text{K}_2\text{Cr}_2\text{O}_7$; Na_2SO_4 - N_2H_4 , H_2O ; quinhydrone at pH 4, and nitric acid and sulfuric acid saturated with oxygen or nitrogen, also 0.1 N- LeSO_4 in N- H_2SO_4 . The theory of the platinum electrode is developed; potentials are shown versus time and pH.—MA. 20122

4. CORROSIVE ENVIRONMENTS

4.3 Chemicals, Inorganic

4.3.2, 6.2.5

Corrosion by Fluoride Solutions. Woodfin E. Shuler. E. I. DuPont de Nemours & Co. U. S. Atomic Energy Commission Pubn., DP-348, Feb., 1959, 15 pp. Available from Office of Technical Services, Washington 25, D. C.

A number of alloys were tested for resistance to corrosion in hydrofluoric acid and nitric acid and mixtures of the two acids. The austenitic stainless steels and the higher chrome-nickel alloys of iron were most promising. Of the materials tested, stainless steel, Type 309Cb, Carpenter 20, Durco D-10, Durimet 20 and Chlorimet 3 were the most resistant to separate solutions of hydrofluoric acid and nitric acid. Each of these alloys showed a significant increase in corrosion rate with increase in temperature. Exposure to nitric acid appeared to passivate Carpenter 20 toward hydrofluoric acid. In the mixed acids, fluoride ion increased markedly the rate of corrosion of all materials tested. The corrosion data indicate that the use of hydrofluoric acid or acidic fluoride solutions will require relatively frequent replacement of dissolver vessels. Stainless 309Cb appeared to be the best of the tested materials considering ease of fabrication, compatibility with the processing of other fuel elements that are dissolved with nitric acid and corrosion resistance. (auth.)—NSA. 18639

4.3.2, 6.3.13, 6.2.5, 6.3.20, 8.4.5

Corrosion Studies for the HRT Chemical Processing Plant. J. L. English, J. C. Griess and D. J. Krause. Oak Ridge National Lab. U. S. Atomic Energy Commission Pubn., ORNL-2735, July 24, 1959, 42 pp. Available from Office of Technical Services, Washington, D. C.

Corrosion resistance of a number of materials considered for use in the Homogeneous Reactor Test Chemical Processing Plant was determined in sulfuric acid solutions. Tantalum was found to be fully resistant to boiling 4 M and 10.8 M sulfuric acid solutions used for the dissolution of corrosion and fission products removed from the homogeneous-reactor fuel solution. Tantalum was immune to hydrogen embrittlement in these solutions. Crystal-bar zirconium and Zircaloy-2 were fully resistant to attack by boiling 4 M sulfuric acid solution but were attacked at rates of 5 to 10 mpy in boiling 10.8 M sulfuric acid solution. Carpenter-20 Cb stainless steel was completely resistant to 4 M sulfuric acid solutions at 38 C, the temperature in the decay storage vessels of the chemical processing plant, and generally was resistant to similar sulfuric acid solutions at atmospheric boiling temperature, approximately 110 C. In a few isolated cases, however, the alloy was prone to stress corrosion cracking and to knife-line attack in several of the boiling 4 M sulfuric acid environments. Corrosion rates of types 347 and 316 stainless steel in boiling 4 M sulfuric acid solution were in excess of 75,000 mpy; however, in the presence of synthetic corrosion and fission products, corrosion attack was reduced to 10 mpy or less. A similar, but less drastic reduction in the rate of attack on Carpenter-20 Cb stainless steel was experienced with additives present in the boiling 4 M sulfuric acid solution. The corrosion rate of the latter alloy in boiling and helium-aerated 4 M sulfuric acid solution was accelerated by ruthenium, a fission-product element. (auth.)—NSA. 19100

4.3.3, 4.2.3

Oil-Ash Corrosion Problem: An Application of the Phase-Equilibrium Approach. W. R. Foster, Research, 12, No. 5, 189-195 (1959).

In oil-ash corrosion sodium, vanadium and sulfur were believed to be the chief

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damaging impurities. Compatibility relations in the system sodium monoxide-sulfur trioxide-vanadium pentoxide were derived on the basis of a simplified phase-equilibrium approach combined with weight-loss experiments and X-ray diffraction evidence. The study showed sodium sulfate, sulfur trioxide, sodium metavanadate, $\text{Na}_2\text{O} \cdot 3\text{V}_2\text{O}_5$, $\text{Na}_2\text{O} \cdot \text{V}_2\text{O}_5 \cdot 5\text{V}_2\text{O}_5$ (or $\text{Na}_2\text{O} \cdot 6\text{V}_2\text{O}_5$), and vanadium pentoxide as potential agents of corrosion.—MA. 19162

5. PREVENTIVE MEASURES

5.3 Metallic Coatings

5.3.2, 8.9.1

How Versatile Application Techniques for Gold Solve Aircraft and Missile Problems. William L. Aves, Jr. *Plating*, 46, No. 11, 1260-1263 (1959).

A low-emissive, highly reflective gold coating is applied to configured aircraft and missile components by the thermal decomposition of a gold resin dissolved in an organic carrier. Immersion gold plating may also be affected by ionic displacement from a commercial solution.—MA. 20168

5.3.4

Role of Hot Dip Galvanizing in Combating Corrosion. (In French.) R. Souske. Paper Presented at 1st European Corrosion Congress. *Corrosion et Anti-Corrosion*, 7, No. 12, 434-439 (1959).

Principal factors which determine the degree of resistance to corrosion of a zinc coating are given as the purity of the zinc, the thickness of coating and the galvanizing temperature. Accelerated corrosion tests, which are described, permit the evaluation of this degree of corrosion resistance and also the relative importance of the three factors determining it. 1 table.—ZDA. 20186

5.3.2

Crack-free and Duplex Chromium Answers to Better Corrosion-Resistance. D. E. Weimer. *Electroplating*, 12, No. 9, 340-343, 348 (1959).

Corrosion-resistance may be increased by the use of crack-free chromium coatings, but even these may crack at thickness more than 0.1 mil. The Duplex chromium process applies first a layer of crack-free chromium from the CF-520 bath, such that no cracking occurs at the high current density areas, followed by a further deposit from the Cr-110 solution to raise the minimum thickness in recesses to at least 0.03 mil. Greatly prolonged corrosion life is claimed for the coating and no redesign of automatic machines is required.—MA. 20238

5.3.4, 6.3.17, 8.4.5

Protection of Uranium by Electrolytic Nickel-Plating and Diffusion. (In French.) G. Chauvin, H. Coriou and J. Hure. *Electrochimica Acta*, 1, 177-189 (1959) July.

An intermediate layer of nickel between uranium fuel element and aluminum sheath protects fuel from corrosion in case of rupture of sheath and from undesirable diffusion between uranium and aluminum. Conditions of production of nickel electrodeposits and diffusion between deposited nickel and underlying uranium. Best diffusion zones are obtained by using a bath containing no

boric acid. Intermetallic compounds formed by diffusion heat treatment of nickel-coated uranium. 32 references.—RML. 20269

5.4.3

Hot-Dip Galvanizing in Continuous Strip Operation. (In French.) J. Lasbleis. *Revue de Metallurgie*, 56, 375-386 (1959) Dec.

Corrosion of steels and cathodic protection of Fe by Zn is discussed. The Sendzimir process facilitates adhesion of the Zn coating because of slight oxidation of the strip, followed by reduction which gives a film of pure Fe. Describes galvanized sheets produced at the Montataire plant.—BTR. 19800

5.3.4, 4.3.2

New Platinum-Clad Steel Resists Attack by Hot Acids. *Machine Design*, 31, No. 26, 36, 38 (1959) Dec. 24.

Pt-5 Rh alloy can now be clad to Carpenter 20 stainless steel. Composite material, developed by Metals & Controls Div., Texas Instruments Inc., is being fabricated in sheet, wire and tubing. Stronger and as corrosion resistant as pure Pt, new composite costs only 1/6 as much. Because ability to stop acid attack at high temperatures (1000 F and above) depends on Pt thickness, clad is available in thicknesses ranging 0.0001-0.005 in. Pt is inseparably clad to stainless by a solid-phase bonding process without use of brazing alloy or other intermediate material. Photomicrograph of sample exposed to 1500 F for one month shows that about 15% of Pt has alloyed with stainless steel. This amount of diffusion is allowable and will not affect acid resistance.—INCO. 19825

5.3.4

On the Deposition of Chromium from Complex Chromium Sulphate Solutions. (In German.) Willi Machu and M.F.M. El-Chandour. *Werkstoffe u. Korrosion*, 10, No. 9, 556-563 (1959).

A survey is made of the best conditions for operating a Cr^{+6} bath of the basic type— $\text{Cr}_2(\text{SO}_4)_3 \cdot 6\text{H}_2\text{O}$ 200, $(\text{NH}_4)_2\text{SO}_4$ 436, urea 240 g/l. It is possible to obtain 10-20% current efficiency for hard and bright deposits, but these have fine cracks. A good adherent coating can be deposited on to an intermediate layer of Ni, Zn, Cu or brass. Trivalent baths are suitable for decorative plating but not for hard Cr since thick coatings from this bath spall. 24 references.—MA. 20096

5.3.4

Bright and Hard Chromizing by French Diffusion Technique. P. Galmiche. *Corrosion et Anticorrosion*, 8, No. 2, 49-58 (1960); *Ingenieurs et Techniciens*, Nos. 5, 6, 51-58, 81-87 (1959).

The ONERA (Office National d'Etudes et des Recherches Aéronautiques) process, based on formation and re-formation of CrF_3 , is described and evaluated. The CrF_3 decomposes at the metal surface, liberating Cr, which diffuses into the base metal, and HF, from which CrF_3 is re-formed on contact with the original chromizing medium (granulated Cr). A bright diffused-Cr layer ~0.1 mm. thick is formed on low-C steel treated for 1½ hr. at 1075-1100 C, or for 3 hr. at 1025-1050 C, longer treatment and higher temperature giving a thicker (> 1 mm) layer. A hard non-bright 20-25-μ layer obtained on C steel after 4-6 hr. at 900-920 C, longer treatment or higher temperature giving no appreciable

increase in layer thickness, can be electropolished. Ni and Co, and alloys based on these, can be chromized under conditions suitable for steels, but Mo W and their alloys usually require a higher-temperature (1400-1600 C) treatment, and the layer can be relatively brittle; satisfactory results are obtainable, however, by precoating with Ni or Fe, followed by bright chromizing. Cu and Cu alloys are unsuited to the process, unless previously coated by chemical or electrodeposited Ni. In general, the diffused layers are completely or highly resistant to attack by corrosive atmospheres or other media, and the process is also applicable to powder metallurgy and to brazing. Structures obtained are illustrated, and general and special applications are discussed.—MA. 20081

5.3.1, 4.2.1, 6.2.5, 6.4.2

Metallic Finishes for Services' Equipment. D. W. Smith. Paper before Inst. Metal Finishing, Symposium on Control in Electroplating, London Branch, Nov., 1958. *Electroplating & Metal Finishing*, 12, No. 9, 331-337 (1959) Sept.

Discusses types of corrosion encountered, protective coating tests and specifications followed by the Services for protection of huge variety of equipment which is stored or used in extremes of climatic conditions all over the world. These climatic conditions are discussed. Problem of vapor corrosion is considered; cadmium is particularly susceptible and Zn, Mg and Pb may also be affected. Examples are shown of severe whisker growth on Ag plating in enclosed electronic assembly (S contaminant liberated from rubber washer); and of pitting corrosion on stainless steel wristwatch back confined to area covered by strap due to lack of air flow. High strength Al alloys, when in form of large extrusions, suffers disastrous form of layer corrosion, which may be further accentuated by galvanic effects from bolted or rivetted-on steel fittings. Principal types of protective metallic finishes used by Services are covered by D.T.D. series of specifications which, although written primarily for aircraft requirements, are being adopted by other Services. Thickness-testing of electrodeposited and sprayed metallic coatings is discussed. Spot tests for identification of cadmium and zinc are outlined.—INCO. 19963

5.3.4

An Outline of the Chemistry Involved in the Process of Catalytic Nickel Deposition from Aqueous Solution. Pts. I, II, III, G. Gutzeit. *Plating*, 46, Nos. 10, 11, 12; 1158-1164; 1275-1278; 1377-1378 (1959).

Pt. I. The author describes electroless nickel baths operating at pH 4-6 & 8-9, respectively; in the acid bath the rate of deposition is almost independent of $[\text{Ni}^{2+}]$ and $[\text{H}_2\text{PO}_2^-]$, but in the alkali bath the rate $\propto [\text{H}_2\text{PO}_2^-]$. Water is required for the catalytic reduction of Ni^{2+} to an amorphous Ni-P alloy; P is apparently supplied from the reduction of H_2PO_2^- by atomic H. It is suggested that H_2PO_2^- is dehydrogenated to PO_2^- ions which combine immediately with water to form HPO_3^{2-} . The formation of H^+ supports the pH dependency of the reactions. 15 references.

Pt. III. The rate of deposition is a catalyst for the dehydrogenation of H_2PO_2^- is metastable at elevated temperature; small amounts of Ni (0.002-0.005 M) cause continuous evolution of H upon immersion of a mild-steel

sample. Pb^{2+} , SeO_3^{2-} , $S_2O_3^{2-}$, CNS^- , and HS^- may reduce or completely inhibit Ni deposition. The rate of H evolution $\propto [H^+]$ and is constant for each pH; plating rate increases with pH.

Pt. III. The rate of deposition is a linear function of the area being plated, and the observed stoichiometry of the reaction was independent of the area. An aqueous solution containing Ni^{2+} and $H_2PO_4^-$ is metastable at elevated temperatures; stability decreases with increasing temperature, $[H_2PO_4^-]$, pH, and concentration of suspended solids. Optimum conditions are tabulated.—MA. 20091

6. MATERIALS OF CONSTRUCTION

6.3 Non-Ferrous Metals and Alloys—Heavy

6.3.20, 4.3.2, 3.8.4

Concentrated Nitric and Dilute Hydrofluoric Acid Mixtures in Dissolution of Zirconium Metal. E. M. Vander Wall and E. M. Whitener. *Industrial and Engineering Chem.*, 51, No. 1, 51-54 (1959).

Dissolution of reactor-grade zirconium plates in mixtures of nitric acid and hydrofluoric acid were determined. Dissolution rates (V) at 40.5 C were determined in mixtures of 0.5M-HF with up to 13M- HNO_3 . HNO_3 concentration has little effect on V, but a high nitric acid concentration is desirable for producing a stable product solution. V (at 40, 50, 60 and 115 C) in 13M- HNO_3 and various concentrations of hydrofluoric acid were determined. The results indicate that initial V is dependent only on the initial hydrofluoric acid concentration in the solution. The activation energy for the dissolution of zirconium in the nitric acid-hydrofluoric acid system is 6.4 kcal/mole from 40 to 115 C.—MA. 19956

6.3.6, 3.2.2, 3.7.4, 3.4.9

Intercrystalline Cracking of Binary and Complex Beta-Brasses Resulting from Moisture in the Atmosphere. A. R. Bailey. *J. Inst. Metals*, 87, No. 11, 380-384 (1958-59).

Binary as well as complex β -brasses undergo intercrystalline cracking in the ordinary atmosphere while under sustained tensile stress of sufficient magnitude. With binary alloys, the tendency increases in composition near the $\beta/(\beta + \gamma)$ phase boundary, becoming very pronounced in alloys actually containing the γ phase. Nevertheless, intercrystalline cracking is still prevalent in alloys lying near the $\beta/(\alpha + \beta)$ phase boundary, although cracking does not occur when the alloys contain sufficient α phase to form an intergranular network. The intercrystalline cracking of both binary and complex alloys takes place only in moist atmospheres, and does not occur in dry argon or in dried air. The effect of moisture is particularly noticeable with binary ($\beta + \gamma$) alloys, which display distinctly better strength and ductility in a dry atmosphere than in the ordinary air. (auth)—MA. 18683

6.3.11, 4.3.5, 3.8.4

Interaction of Oxygen and Hot Gold. Pt. I. L. G. Carpenter and W. N. Mair. *Trans. Faraday Soc.*, 55, No. 11, 1924-1936 (1959).

Reaction between oxygen at 3 μ mercury pressure and a gold filament at

~900 C was studied by the removal of oxygen and the evaporation of gold. It is shown that oxygen oxidizes impurities on the gold surface and allows the metal to evaporate at an increased rate. Active oxygen is formed on the gold surface, and after leaving the metal surface it impinges on the reaction-vessel wall. If the walls are covered with evaporated gold, then mol. oxygen is returned to the gas phase, but if the walls are not so covered then the active oxygen reacts with materials present and is removed from the gas phase. In an Appendix it is shown that the activated species is probably $^1\Delta$ electronically excited oxygen mol. The structure of evaporated gold films is discussed in a second Appendix. 31 references.—MA. 20285

6.3.14, 4.4.4, 4.4.2, 5.8.4

Corrosion of Tinplate by Victoria Plum Syrup. F. W. Salt and J. G. N. Thomas. *J. Iron & Steel Inst.*, 188, Pt. 1, 36-45 (1958) Jan.

Study of corrosion of steel by Victoria plum syrup revealed that the syrup contains both inhibitors and accelerators of corrosion. Stannous tin inhibits corrosion appreciably but stannic tin slightly accelerates it. A number of possible organic inhibitors were investigated in plum syrup and 2, caprylic and caproic acids, were found to give appreciable inhibition in presence of 25 ppm stannous tin. Caprylic acid alone in plum syrup greatly increases steel corrosion rate. Chrysanthemin, the principal coloring matter of Victoria plums, was shown to inhibit both cathodic and anodic processes in corrosion of steel in malic acid, but to have no significant effect on corrosion of steel or steel-tin couples in Victoria plum syrup. Graphs, tables, 16 references.—INCO. 17683

6.3.15, 3.7.2, 3.7.4

On the Corrosion-Resistance of Titanium Alloys. Pt. III. The Equilibrium Diagram of the Titanium-Palladium System. (In Japanese.) Hideo Nishimura and Tsuyoshi Hiramatsu. *J. Japan Inst. Metals (Nippon Kinzoku Gakkai-Si)*, 22, No. 2, 88-91 (1958).

Metallographic examination, X-ray analysis and melting point determination were carried out over the whole range of compositions. One intermediate compound, $TiPd_3$, was identified, of hexagonal D_{3h} -type structure ($a=5.489$, $c=8.964$ A, $c/a=1.635$). A eutectic reaction occurs at 47% palladium and 1080 C, a peritectic reaction at 89% palladium, and a eutectoid reaction at approximately 25% palladium and 735 C. The solubility of palladium in β -titanium is approximately 45% at 1080 C and of palladium in α -titanium approximately 5% at 735 C. That of titanium in γ -palladium is 11% at 1000 C.—MA. 17506

6.3.5, 5.3.4, 3.5.9

Research for Coatings for Protection of Niobium Against Oxidation at Elevated Temperatures. Period covered July 1, 1957 to August 31, 1958. Emanuel C. Hirokis. Horizons, Inc. U. S. Wright Air Development Center, Technical Report 58-545, Sept. 12, 1958, 73 pp.

Niobium alloys were prepared offering up to 20 fold reduction in oxidation at 2000 F compared with pure niobium. Flame sprayed and electrodeposited coatings were developed giving more complete protection for 4 to 6 hrs. at 2500 F. Preparation of alloys and coatings together with test procedures and results are described for both phases. (auth)—NSA. 18776



J. M. PEARSON

Contributions of J. M. Pearson to Mitigation of Underground Corrosion

Six articles by the late Dr. J. M. Pearson and one by an associate prepared under his leadership are reprinted for the information of new workers and established investigators into underground corrosion problems. Dr. Pearson, recipient of the 1948 NACE Speller Award for achievements in corrosion engineering, is recognized for his outstanding work on problems associated with corrosion of metals underground. The articles, originally published during 1941-44 in *The Petroleum Engineer* and in *Transactions of The Electrochemical Society* are reprinted by permission in this book dedicated to him.

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Topic of the Month

Deep Well Ground Beds for Cathodic Protection Of Multiple Lines in Highly Congested Industrial Areas*

By J. R. GATES*

Introduction

THREE COMPANIES have multiple lines on the same right-of-way through approximately eight miles of the highly-congested Hammond industrial area. These firms are Service Pipe Line Company, Sinclair Pipe Line Company and American Oil Company (formerly Standard Oil Company, Indiana).

Because of leaks on the Sinclair and American lines in 1959, a joint cathodic protection system, long talked about, was given renewed impetus. Several conferences were held by representatives of the three companies to develop a suitable work plan.

* Submitted for publication March 16, 1961.

* District Engineer, Joliet District, Service Pipe Line Co., Joliet, Illinois.

Abstract

An account is given of the successful installation of deep well type high silicon iron anodes in a highly congested industrial area. Multiple lines were cathodically protected in this way despite the presence of nearby gas and water mains, power line grounds and underground telephone cables. A special control system was designed for installation at each rectifier, involving use of switches, ammeters, rheostats, non-reversing current diodes and surge protectors. 5.2.1

Since the Service and American lines were broken into five sections with insulating flanges, five rectifier sites were chosen and current requirement tests run. When insulating flanges were shorted, heavy stray currents built up in all lines. But with insulating flanges open, it was found that one to two amps per line would afford suitable protection to both the Service 14-inch and 18-inch lines and the American 8-inch and 12-inch lines. The two Sinclair 8-inch lines and 22-inch line did not have insulating flanges in this area. Because of this and several shorted casings, it was impossible to fully protect these lines, although some pro-

tection was afforded. These preliminary tests were run with temporary ground beds of aluminum foil and rock salt at the five proposed rectifier sites. Sinclair Pipe Line Company provided the test current with a convenient d-c generator.

Space was not available at any site for conventional ground beds and this factor, plus the probability of intense interference with the huge network of gas and

TABLE 1—Resistance Values in Drilling Operation

Depth, Ft.	Drill String To 14-Inch Line, Ohms	Drill String To Remote Earth, Ohms (C = 300', P = 150')
23.....	2.00	1.65
33.....	1.88	1.50
43.....	1.65	1.30
53.....	1.55	1.20
63.....	1.40	1.05
73.....	1.55	1.08
	(set 63' of casing)	
83.....	1.23	0.86
93.....	1.15	0.82
103.....	1.10	0.80

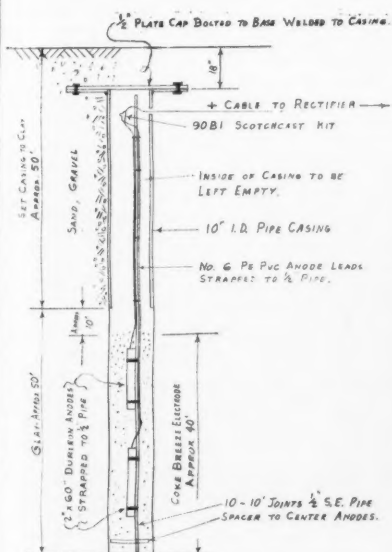


Figure 1—Installation sketch of deep well ground bed.

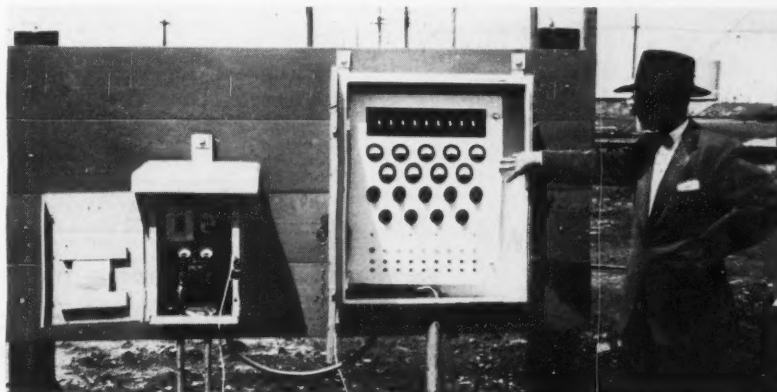


Figure 2—Control panel for multiple line deep well protection system.

water mains, house services, power line grounds, underground telephone cable and foreign pipe lines prevalent in this area, influenced strongly the decision to use deep well ground beds.

Anode Installation

Figure 1 shows a cross section of the deep wells. Because of the presence of limestone underlying the area at a depth of 100 to 150 feet, it was decided to drill each well to approximately 100 feet. Since each ground bed would be required to drain only 10 to 15 amps, a standard design was adapted of two high silicon iron anodes in each bed on about 20 foot

centers with 40 feet of coke breeze poured and tamped around the anodes. This was installed in a dry hole. Typical resistance values achieved as measured during the drilling operation are given in Table 1.

Because of the diverse current requirements for the seven lines, it was decided that a rather elaborate control system should be designed for installation at each rectifier. This included a separate switch, ammeter, rheostat, non-reversing current diode and surge protector on the lead to each pipe line (see Figure 2). The many manhours saved from convenient rapid adjusting has more than paid off the original investment.

The many minor interference problems caused by these units in a highly congested area have been or are being worked out with the companies involved through the efficient coordination of the Chicago Region Committee on Underground Corrosion (T-7B).

Conclusion

It is the conclusion of those involved in this installation that in a highly congested area where conventional ground beds could not be used, the deep well type have made possible a successful system of cathodic protection at a reasonable cost.

**Any discussion of this article not published above
will appear in December, 1961 issue.**

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Pitting Corrosion by Water Flood Brines*

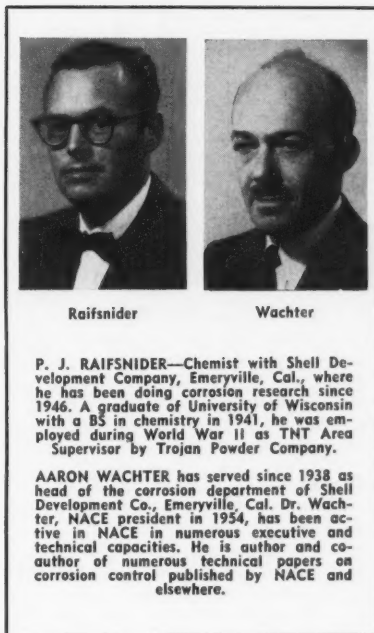
By P. J. RAIFSNIDER and A. WACHTER

Introduction

SERIOUS CORROSION in the form of internal pitting of steel injection lines has been suffered in several closed system (anaerobic) type water floods. In one system, as many as 200 pipe leaks from this cause occurred in one month. General corrosion in these cases is negligible or of minor importance with respect to wall penetration. Leak frequency tends to increase with time despite liberal use of various corrosion inhibitors. Exposure of conventional strip type corrosion specimens of steel in lines of these floods usually shows low rates of general attack and rarely manifests the serious pitting concurrently occurring in the injection lines. Laboratory tests made using the conventional rotating bottle technique, give results similar to those observed on specimens exposed in the field. Good commercial corrosion inhibitors usually give excellent results in reducing general attack in these tests, but substantially no pitting occurs with or without inhibitor. Since pH of these waters is usually about 6, acidic corrosion is considered to be the cause of the mild general attack observed in such laboratory tests.

Early in the investigation of the pitting problem, oxygen was suspected as the responsible corrosive agent. However, polarographic analyses of injection water usually show that oxygen content is generally less than 0.4 ppm (limit of precision of this analysis). This would be taken to indicate that oxygen was not a major corrosion factor. Then, the possibility that scale deposits might be interfering with inhibitor action was considered. Following this line of thought, badly scaled injection lines were acidized and hydrochloric acid was added continuously to the injection water to maintain sufficiently low pH to prevent further scale accumulations. Inhibitors showing greater effectiveness in laboratory weight loss corrosion tests were substituted for those previously used. These treatments resulted in only temporary improvement. Leak frequency was again high after only several months operation under these new conditions.

In further consideration of this pitting corrosion problem, attention was focused on the possibility that trace oxygen contamination was the primary causative agent despite low analysis values. It is well known that scale and dirt deposits provide anodic sites for operation of differential oxygen concentration cells. The areas under these deposits are par-



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AARON WACHTER has served since 1938 as head of the corrosion department of Shell Development Co., Emeryville, Cal. Dr. Wachter, NACE president in 1954, has been active in NACE in numerous executive and technical capacities. He is author and co-author of numerous technical papers on corrosion control published by NACE and elsewhere.

tially shielded from rapid replenishment of oxygen and thus become anodic to surrounding areas. Cells of this type cause serious pitting corrosion in operation of boilers unless measures are taken to insure complete oxygen removal from boiler feed water. In addition, the character of the pitting with boiler water containing trace amounts of oxygen appears similar to that experienced in several closed system water floods. Even with only trace concentrations of oxygen (e.g., 0.1 ppm or less), the large volumes of brine moving through the lines would make significant amounts of oxygen available to large cathodic areas surrounding very small anodic spots. Solid corrosion products accumulating at such anodic sites would enhance the shielding from oxygen and thus aid in persistence of the concentration cell. Also, the inhibitor carried by the brine is hindered from free access to these anodic sites. This analysis could account for the rapid, widely distributed pitting experienced in water flood injection lines.

In order to study the factors responsible for such corrosion, there was essential need for an experimental method that would demonstrate both pitting and general corrosion attack of steel in water flood brines. In addition, unless an effective test could be developed it would be impossible to assess remedial measures in the laboratory and follow the behavior of

Abstract

Serious pitting corrosion has been experienced in several closed system water floods in secondary recovery operation in oil fields. A laboratory test method was developed which rapidly reveals pitting tendencies in samples of water flood brines. Using this method, it was shown that trace amounts of oxygen are responsible for the pitting in these water floods, and that corrosion can be prevented by adding an oxygen scavenger such as sodium sulfite. Relatively low concentrations of corrosion inhibitor are adequate when oxygen is completely removed. In addition, a low cost, rapid response test probe for field use has been developed to aid in monitoring mitigation treatments. 3.2.2, 4.6.9

subsequent recommended treatments in the field.

A laboratory test method was then developed which rapidly reveals pitting tendencies of water flood brines. This method utilizes an artificially induced crevice created by shielding a small part of the surface of a clean steel specimen with an inert material. This device simulates the condition that can occur in the field wherein a differential concentration galvanic cell is created, the small shielded area being strongly anodic to the surrounding metal surface. In practice, these areas could occur in pipe lines through scale or dirt deposits.

Experimental Technique

In the laboratory study of factors controlling this pitting type of corrosion, use was made of an electrical resistance cor-

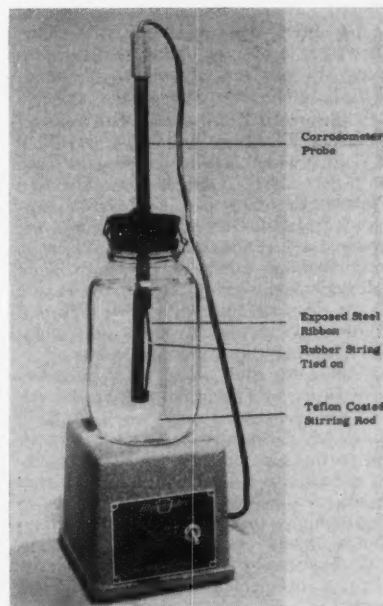


Figure 1—Laboratory corrosion test assembly.

* Submitted for publication December 5, 1960. A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, Tulsa, Oklahoma, Oct. 25-28, 1960.

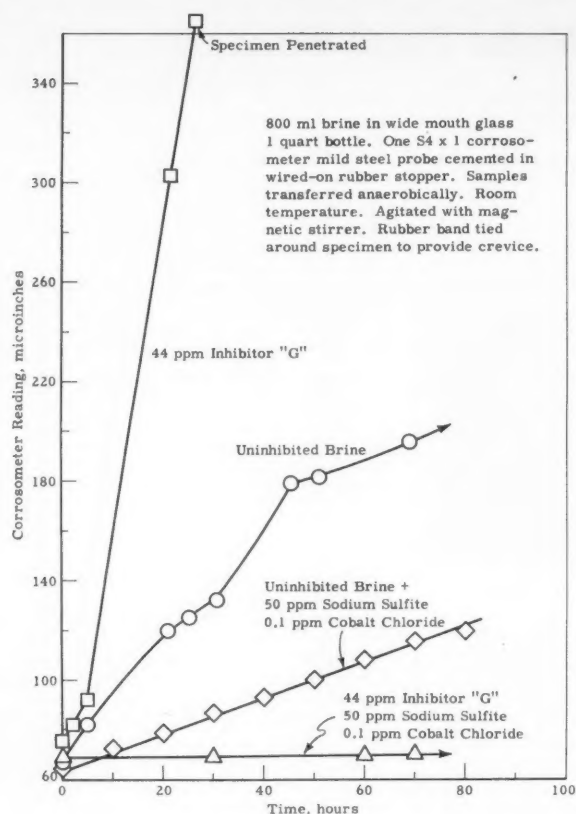


Figure 2—Corrosion by flood water "A".

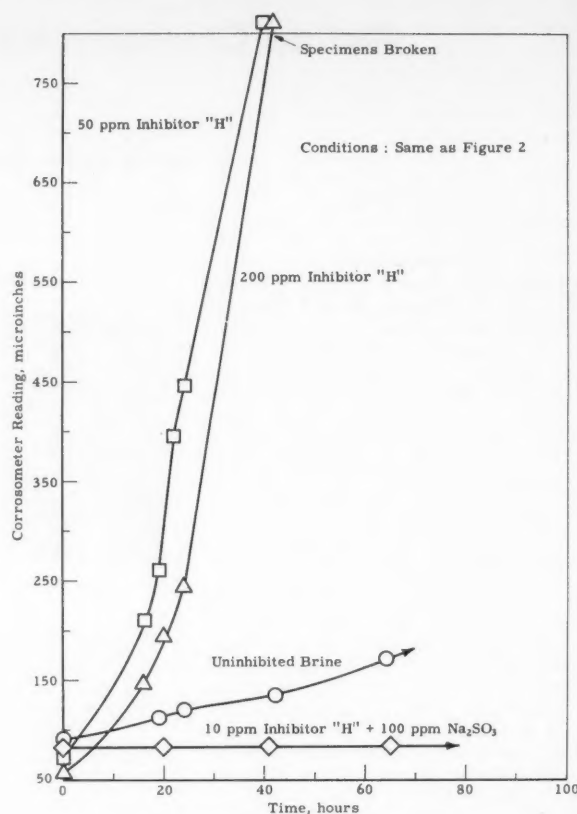


Figure 3—Corrosion by flood water "B".

rosion measuring device. This method is based on measurement of the electrical resistance of a ribbon, wire or tube of the test metal. When the specimen corrodes, it becomes thinner and its electrical resistance increases. From this increase, one can determine the loss of metal from corrosion. It is possible, therefore, to observe or measure continuously the progress of corrosion without disturbing the environment or specimen.

The corrosion probes used had exposed elements of mild steel ribbon 4 mils thick x 1/8-inch wide x 2 3/4-inches long. A piece of thin rubber band was tightly tied around the center of the ribbon to provide an artificial crevice which could act as an anode site for a differential oxygen concentration cell. Rubber bands were selected over cotton string or plastic bands since the use of rubber afforded continuous tension around the metal strips during the corrosion process. Corrosion meter dial readings (microinches penetration) reflected both general attack and also the pitting under the band and, therefore, had no direct quantitative corrosion rate significance, being useful only as a qualitative indicator. The very thin 4 mil ribbon was needed in order to obtain high sensitivity of meter readings and to avoid consumption of all available oxygen by corrosion before large changes in element resistance had occurred.

The probes were exposed in one quart

glass bottles nearly filled with brine samples taken anaerobically in the field. The transfers from the field sample containers to the laboratory test bottles were made in a nitrogen filled chamber so as to minimize oxygen contamination. The corrosion tests were made at ambient temperature (about 70 F) and agitation was provided using magnetic stirrers. The probes were inserted through rubber stoppers which were subsequently securely wired into the glass bottles. As additional precaution against adventitious oxygen leaks, all connections were sealed with Permatex No. 2 gasket cement. The completed corrosion test assembly is illustrated in Figure 1.

It was recognized that field samples of brine probably suffered additional contamination with air before arriving at the laboratory. This was not serious provided gross aeration had not occurred, since emphasis in the present study was on revealing factors responsible for pitting and methods for control of this type of corrosion. Effects of oxygen, as explained later, were shown by chemical removal of oxygen and its subsequent addition.

Corrosiveness of Field Water Samples As Received Samples

Results of laboratory studies made with uninhibited injection water as received from floods "A" and "B" are illustrated by one of the curves in Figures 2 and 3.

Pronounced pitting was not found with either water sample; however, general corrosion was somewhat high (18 mils/yr) in flood water "A" and moderate (5 mils/yr) in flood water "B". The initial oxygen content of these waters was not measured, but the samples were handled anaerobically to minimize additional oxygen contamination.

Effect of Organic Inhibitors

The effects of adding various commercial organic inhibitors to the injection water samples from "A" and "B" fields are shown in Figures 2 and 3. The brine "A", inhibited with 44 ppm Inhibitor "G" (long chain cyclic amine), showed severe pitting under the rubber band, resulting in specimen penetration in less than 24 hours. The water sample "B", inhibited with either 50 or 200 ppm Inhibitor "H" (water soluble imidazoline), also showed very severe pitting, resulting in specimen penetration in approximately 30 hours. The appearance of the specimens showed that general corrosion was reduced in the presence of the inhibitors; however, pitting was sharply accelerated. There was no significant difference in results with several common water flood corrosion inhibitors (see Table 1). Acceleration of pitting by the added inhibitor may be attributed to the inhibitor maintaining a generally more cathodic condition on exposed steel surfaces to which it is readily available as compared to

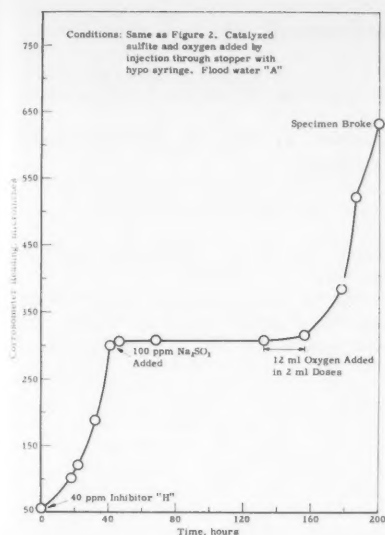


Figure 4—Effect of oxygen on pitting corrosion.

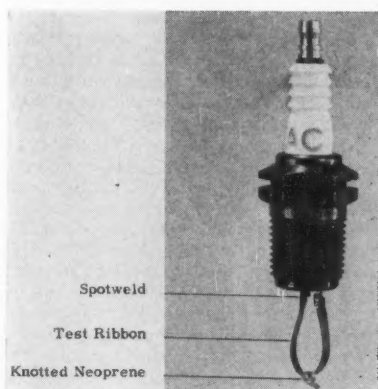


Figure 5—Spark plug probe for field tests.

small shielded areas to which free access of inhibitor is hindered.

Effect of Oxygen Scavenging

The effects of chemically removing oxygen from these field water samples are illustrated in Figures 2 and 3 and Table 1. Oxygen scavenging was accomplished by using cobalt catalyzed sodium sulfite. In the "A" flood water, 50 ppm sodium sulfite with 0.1 cobalt chloride was sufficient to remove all oxygen present and provide a slight sulfite residual. The water samples from "B" flood appeared to contain a considerably higher concentration of oxygen and 100 ppm of catalyzed sodium sulfite was used.

In the uninhibited samples, oxygen removal resulted in elimination of pitting and slight reduction of general attack (see Figure 2).

With samples inhibited with various proprietary materials, oxygen removal resulted in complete inhibition. No general corrosion and no pitting were observed. This was true even at inhibitor

TABLE 1—Inhibitor Evaluations*

Flood Water	Inhibitor	Inhibitor Conc., ppm	Na ₂ SO ₃ , ppm	CORROSION	
				Pitting	General
"A"	"H" (Water soluble imidazoline)	200	0	+++	0
		40	0	+++	0
		50	0	0	0
		20	50	0	0
		10	50	0	0
"A"	"G" (Cyclic amine)	200	0	+++	0
		20	50	0	0
		10	50	0	0
"B"	"H" (Water soluble imidazoline)	200	0	+++	0
		100	0	+++	0
		20	100	0	0
		10	100	0	0
		5	100	0	0
"B"	"L" (Coco amine acetate)	150	0	++	0
		10	100	0	0
		5	100	0	+

* Conditions: As given in Figure 2. +++ = complete penetration of probe ribbon, ++ = severe attack, + = moderate attack, 0 = no attack.

concentrations as low as 10 ppm (see Table 1). There were no marked differences in effectiveness among the various inhibitors tested.

In order to demonstrate further the effects of oxygen on pitting corrosion, an experiment was made providing for oxygen scavenging after deep pitting had occurred on a rapidly corroding probe. Results of this experiment are given in Figure 4. A crevice containing probe was exposed in brine "A" inhibited with 40 ppm Inhibitor "H". Rapid corrosion occurred. At 40 hours, when the probe was approximately one-half penetrated, 100 ppm of catalyzed sodium sulfite was anaerobically injected into the system. In a short time, corrosion completely ceased. After 90 hours with no corrosion occurring, 12 ml of oxygen was added in 2 ml portions (about 16 ppm). Rapid corrosion resumed, resulting finally in specimen penetration. This experiment is significant since it indicates that complete oxygen removal will halt pitting that has progressed to a serious degree. In addition, it shows that re-contamination with oxygen will cause the localized corrosion to proceed again.

The conclusions from the laboratory studies which included many additional confirmatory experiments can be summarized as follows:

1. Trace oxygen, no inhibitor = some general, some pitting attack.
2. Trace oxygen + inhibitor = little general, severe pitting attack.
3. No oxygen + no inhibitor = some general, no pitting attack.
4. No oxygen + inhibitor = no general, no pitting attack.

It seems clear that oxygen must be rigorously excluded from water flood systems that are suffering severe pitting attack. If oxygen is present, even massive doses of inhibitor (200 ppm which would be economically prohibitive) are ineffective in halting pitting. It does not necessarily follow that pitting corrosion will be encountered in a given flood system as the result of trace contamination with oxygen if the water naturally is capable of consuming rapidly, by chemical or

biochemical reaction, all the oxygen inadvertently entering the system.

Field Studies

In order to establish the validity of the conclusion that trace oxygen contamination is responsible for pitting corrosion in flood waters, it was recommended that catalyzed sodium sulfite be added to two floods that were experiencing numerous pipe leaks per week. In one of these floods, removal of oxygen from the injection water reduced the corrosion failures from approximately 20 leaks per week to one per year. In the other flood, the response to oxygen scavenging was less dramatic. The situation in this flood was considerably more complicated since serious corrosion had occurred over a much longer period; consequently, much of the existing injection piping was severely damaged. However, the leak frequency rate has been steadily declining. This has been accomplished with corrosion inhibitor concentrations at one half the values maintained before oxygen scavenging.

Field Test Probe

It is recognized that one of the difficulties concerned with water flood operations has been the lack of a sensitive field corrosion test able to detect pitting in a short time period. Usually, weight loss corrosion specimens have to be exposed for several weeks to get results of significance, even when a shielded area (use of neoprene rubber band) is provided to reveal pitting. Since brief operational changes or upsets causing rapid corrosion could occur during this period, it is difficult to relate operating factors to corrosion.

To meet this need a specimen unit was designed that serves as a convenient, rapid response indicator of severe pitting conditions. This unit is illustrated in Figure 5 and was made in the following manner. One end of a steel ribbon (2 inches long, 1/8 inch wide, 4 mils thick) was spot welded to the center electrode and the other end to the ground electrode of a Model-T type sparkplug* (so

* The spark plugs must be tested in advance to insure that those used do not have high internal resistance.

selected because the threads are $\frac{1}{2}$ inch IPS and thus can be fitted easily into existing piping systems). An artificial crevice is made on the strip by tying a piece of neoprene rubber band around it. This crevice sensitizes the strip to pitting. Rapid penetration of the strip under the rubber band should occur if trace oxygen contamination exists, thus breaking the specimen. The integrity of the specimen can be checked easily without removal by a simple circuit tester such as a one cell flashlight battery connected to a flashlight bulb or to an ammeter. Connecting one tester lead to the pipe and the other to the center electrode of the plug indicates whether the specimen

has parted or is still intact. Field trials of these specimens have shown them to be quite useful.

DISCUSSION

Question by Charles C. Nathan, The Texas Co. Research Lab., Bellaire, Texas:

What was the effect of small amount of oil on the efficiency of the organic inhibitors in the test system?

Reply by P. J. Raifsnider and A. Wachter:

We did not test in the presence of small amounts of oil. One would pre-

sume the efficiency of most organic inhibitors would improve with a significant amount of oil in the injection brine. We do, however, try to avoid the carry-over of crude oil into the injection water.

Question by Ray W. Amstutz, Tulsa Oklahoma:

How long does it take to get failure of the spark plug test specimens?

Reply by P. J. Raifsnider and A. Wachter:

This of course depends upon the corrosiveness of the injection water. We have had failures in as little as 3 days.

Any discussion of this article not published above will appear in December, 1961 issue.

NACE Guide for Preparation of Articles for Publication

Persons interested in submitting articles on corrosion for publication in CORROSION can obtain upon request a copy of the "NACE Guide for Preparation of Papers." Write to CORROSION, National Association of Corrosion Engineers, 1061 M & M Bldg., Houston 2, Texas.

The Casing Inspection Tool — An Instrument for the In-Situ Detection Of External Casing Corrosion in Oil Wells*

By T. R. SCHMIDT

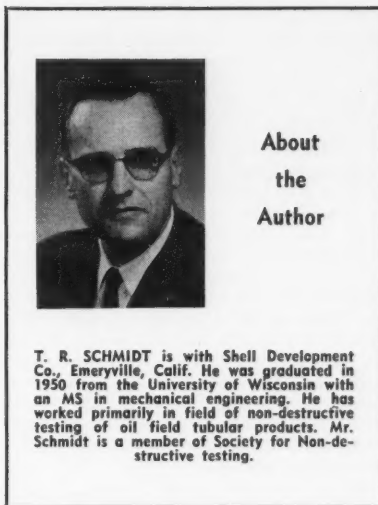
Introduction

THE CASING inspection tool is a down hole instrument developed for the detection of external corrosion in oil well casings. It is a surface recording, non-contacting, eddy-current instrument run in casing on a single conductor wire line. It measures and records casing wall thickness regardless of whether metal loss is on the inside or outside of the pipe. In those instances where it is not well established whether corrosion is primarily external or internal, it may be necessary to run an internal caliper in conjunction with the instrument.

Fortunately, casing corrosion does not seem to be a widespread problem. Where it does exist, however, it is often a very costly problem. Working over a well for the detection and repair of casing leaks is a major expense. Casing corrosion will become of increasing importance as existing wells become older and will be accentuated by some secondary recovery schemes which may demand original strength and soundness of an older well.

Until now no method short of pulling the casing has been available to detect external corrosion before leaks develop. Electrical potential profile surveys taken in wells may show where corrosion is likely to be occurring but give no indication as to the extent of damage that has been done. Often external corrosion is not suspected until the first leak appears and by that time corrosion damage can be extensive. Included in this damage, while difficult to estimate, should be the effects of product loss to higher thief zones and the effects of producing formation wetting. Undoubtedly minor corrosion is present in most wells. The important question is will it play a significant part in the overall economic picture of the field? If corrosion could be detected in its earlier stages, field plans and policies could be made taking it into account. In practice this detection process might consist of periodically surveying a few representative wells in a field to determine if corrosion damage is present and if so, at what rate it is growing.

The lack of a test for external casing corrosion has resulted in relatively little effort to control this type of attack. Down hole conditions with respect to mineral waters, bed conductivities, stray currents, etc. are difficult to simulate in the laboratory. On the other hand full scale tests are generally too slow in that a criterion for success of such a test might be no leaks in, say, 20 years. Numerous corrosion alleviation schemes have been pro-



About the Author

T. R. SCHMIDT is with Shell Development Co., Emeryville, Calif. He was graduated in 1950 from the University of Wisconsin with an MS in mechanical engineering. He has worked primarily in field of non-destructive testing of oil field tubular products. Mr. Schmidt is a member of Society for Non-destructive testing.

posed from time to time such as cathodic protection, corrosion resistant materials, plating, protective coatings, inhibited muds and cements, full string cementing, etc., but have yet to be fully evaluated. Cathodic protection is of particular interest in that it can be applied at any stage of a well's life. There are certain questions with regard to the currents necessary for protection and the effects of stray currents that are not completely answered.

Corrosion pits which cannot be broken down by hydrostatic testing go undetected during well workovers. Pits which could develop into leaks in a few months go unrepaired. In general very little is known about down hole leaks or other weaknesses. Often the only information available is that the casing will not hold pressure in an interval straddled by packers. This could be caused by one large hole, several small ones, split casing, drill pipe wear, etc. Unfortunately, testing methods used in the past seldom pointed to type of leak involved.

Potential uses of the casing inspection tool can be summarized as follows:

1. Periodic survey of a sampling of wells to determine if casing corrosion is present and if so, at what rate it is growing.
2. Evaluating protective measures.
3. Locating and determining the character of leaks, pits and other weaknesses prior to repair.

Choice of Inspection Technique

Several basic inspection schemes of casing wall thickness measurement such as ultrasonic or the coil impedance an-

Abstract

A surface recording, down hole instrument for the detection of external corrosion in oil wells is described. The tool is a non-contacting, eddy-current instrument which measures and records casing wall thickness. Equations are presented which are useful in describing the tool's action. Examples of laboratory and down hole logs are shown demonstrating the tool's response to general thinning, pits, holes, collars, etc. Results of a number of down hole runs representing some 40,000 feet of well logged are discussed. 2.4.2, 8.4.3

alyzing form of eddy-current instrument were considered for this down hole inspection problem. The remote field analyzing form of eddy-current testing chosen, while relatively little used, offers certain advantages for this particular application. Some of these are:

1. The tool is non-contacting. Clearances of from 5 percent to 10 percent of the casing inside diameter are used.
 2. The tool is unaffected by dirt or scale on the walls of the pipe.
 3. Position of the tool with respect to centering or canting has little effect on the records.
 4. The record or log obtained is linear with respect to average wall thickness with an exaggerated indication of flaws or discontinuities superimposed on this basic indication.
 5. Full 360 degree scanning of the pipe is obtained.
 6. Logging speeds up to 3600 ft. per hour can be used with 1800 ft. per hour commonly used.
 7. The tool operates over the single conductor type logging cable.
 8. The operation of the tool is independent of the fluid in the hole.
- The bases for these advantages are developed in subsequent paragraphs.

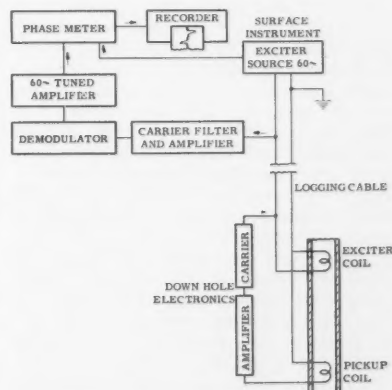


Figure 1—Block diagram casing inspection tool.

* Submitted for publication January 9, 1961. A paper presented at the 17th Annual Conference, National Association of Corrosion Engineers, Buffalo, New York, March 13-17, 1961.

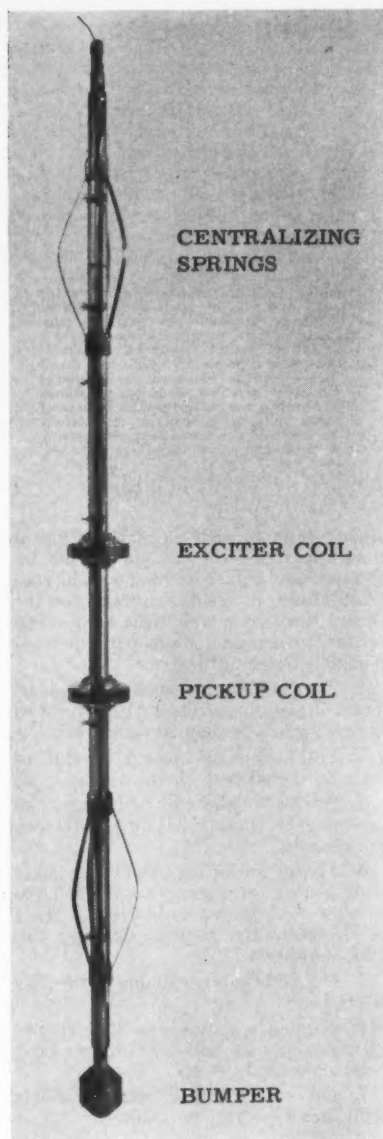


Figure 2—Down hole tool.

Description

A block diagram of the instrument is shown in Figure 1. An exciter coil fed from an alternating current source at the surface sets up eddy-currents in the pipe walls. The effects of these eddy-currents on the magnetic flux distribution are detected by a pick-up coil. The exciter and pick-up coils are so spaced that there is in effect no direct coupling between them. Any coupling between the coils is done by flux which has for practical purposes penetrated the walls of the pipe and has been attenuated and shifted in phase according to the classical eddy-current¹. Because of heavy attenuation of flux penetrating the pipe walls, the pick-up signal is quite small and must undergo substantial amplification before it is transmitted back to the surface as modulation of a 3 kc carrier. In the surface instrument this carrier is filtered off the line,

which it, of course, shares with the exciter signal, and is amplified and demodulated. At this stage the signal is a reproduction of the pick-up coil signal but in the surface instrument. The signal is further amplified in a tuned amplifier stage, and its phase lag or time lag with respect to the original exciter signal determined in a recording phase meter. It is this record of phase lag of the pick-up signal with respect to the exciter signal which is the log of casing wall thickness. Theory indicates that the phase lag of this pick-up signal should be linearly related to wall thickness. In the instrument a differential pick-up coil is used to give an accentuated indication of flaws, pits, etc. which is superimposed on a linear average wall thickness indication.

Figure 2 is a picture of the down hole instrument. Centralizing springs are used to give the instrument equal sensitivity in any direction and to minimize coil wear. Coil clearances of from 5 percent to 10 percent of the pipe inside diameter are commonly used. One set of coils is sufficient for the various weights of pipe in a given nominal casing size, but a set of coils is required for each nominal casing size. The instrument is constructed to withstand the high pressures and temperatures normally encountered in oil wells. The down hole electronics are carried within the hollow mandrel on which the coils are mounted.

Figure 3 is a view of the down hole electronics. The unit is built to an outside diameter of 1.125 inch and is fully transistorized and battery powered. The units are checked out for operation to 325 F. Maximum reading thermometers are carried.

Figure 4 shows the surface instrument. Phase angle is measured by a servo-driven phase shifter which nulls the pick-up signal with respect to the exciter. A linear output voltage as a function of phase angle is available from the instrument for recording.

Theory

The classical eddy-current equations are as follows:

$$B = B_0 e^{-2\pi d \sqrt{\frac{f\mu}{\rho \times 10^9}} \sin \left(2\pi ft - 2\pi d \sqrt{\frac{f\mu}{\rho \times 10^9}} \right) \quad (1)$$

where:

B = magnetic flux density at depth (d)
 B_0 = magnetic flux density at surface
d = depth (cm)
f = frequency in cycles per second
 μ = relative permeability
 ρ = resistivity in micro-ohm cm
t = time in seconds

In the equation, which is, incidentally, of the same general form as that for cyclic heat flow in one dimension, the amplitude variation of flux density with depth is given by:

$$\text{Amplitude (B)} = B_0 e^{-2\pi d \sqrt{\frac{f\mu}{\rho \times 10^9}}} \quad (2)$$

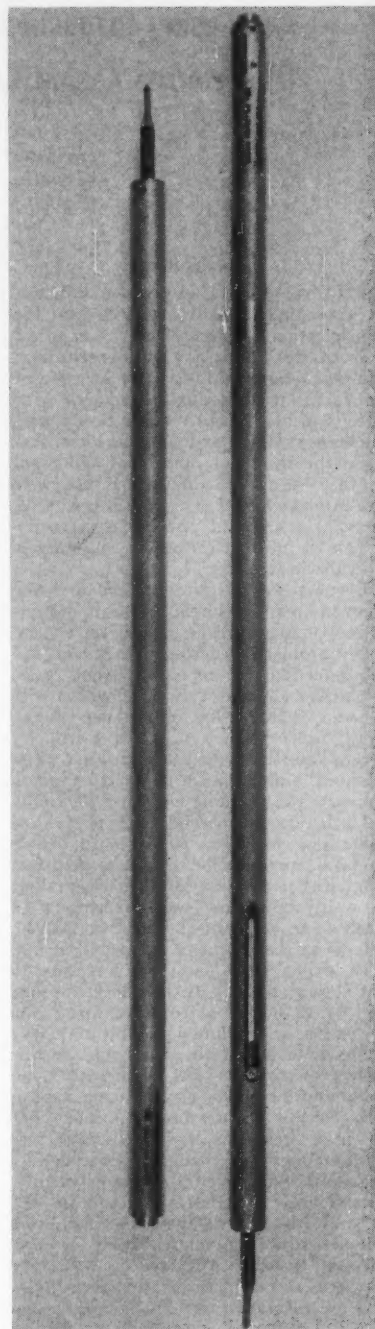


Figure 3—Down hole electronics.

and phase shift by:

$$\text{Phase shift } (\phi) = 2\pi d \sqrt{\frac{f\mu}{\rho \times 10^9}} \quad (3)$$

Either variation in amplitude or phase shift can be used as a measure of wall thickness (d). Because of the linear relation between phase shift and wall thickness as well as a reduction in telemetering problems, it was chosen over amplitude.

The equation (1) has been derived for a semi-infinite conducting medium

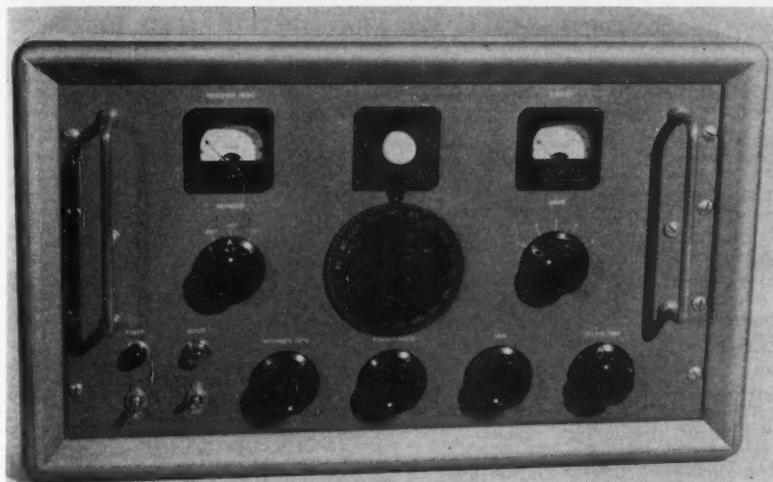


Figure 4—Surface instrument.

bounded only by the plane $d=0$ and with the magnetic flux density at the surface parallel to this plane. It has been found to apply in many instances to wires and cylinders². The flux distribution resulting from an exciter coil inside a pipe is more complex than under the assumptions which these equations were derived. In addition the voltage being developed by the pick-up coil is an integrated effect of the flux being intercepted by it. To substantiate, however, that equation (1) is useful in describing the tool's action, a curve, Figure 5, of phase lag of the pickup signal with respect to the exciter as a function of wall thickness was determined. The agreement of the measured points (circled) and an average straight line through them is shown.

As further substantiation, a curve of phase lag as a function of frequency was determined (Figure 6). Equation (1) indicates that the phase lag should be proportional to the square root of frequency (a parabola). A true parabola adjusted to pass through the measured phase shift point at 60 cycles is shown along with the measured points (circled). While the agreement is not as close as in the previous curve, the deviation is not excessive.

In the actual instrument the phase of the pick-up coil voltage, after telemetering to the surface, is measured with respect to the exciter voltage at the surface. These voltages are not in phase with their respective flux fields down at the tool whose behavior can be described by equation (1). The phase shifts along the signal path and phase angle between voltage and flux are fixed however, and variations in phase angle between these surface voltages will duplicate the variations in phase angle of the flux fields at the tool. As an example, in calibrating the instrument, a reading is first taken in air which is the equivalent of being in a pipe of zero wall thickness ($d=0$). While the instrument will read some phase shifts with respect to the exciter and pick-up voltages, this reading is considered the zero reading. The tool is then inserted into a pipe of known wall thickness and the scale determined from the

difference of the new reading from the zero reading. The increase in phase lag is due to the presence of the pipe walls.

Casing Logs

Much of the development work on the tool was done using a 30-foot section of 7-inch casing in the laboratory. This casing had machined intervals of known outside diameter reduction on one half and simulated "flaws" on the other. It had a standard screwed collar connection in the middle. A log of this test pipe and its cross section are shown in Figure 7. Thinning is to the left. Note that for any one boundary, as in the turned down sections, there is a double break in the log. Half the break occurs as one coil passes the boundary and the deflation is completed when the second coil passes the boundary. This effect can be seen to a lesser extent on the flaws and collars. The sharp break is due to the pick-up coil passing the flaw and the duller break due to the exciter.

There are two saw cuts on the casing sample labeled transverse cut and longitudinal cut. These are each about 3 inches long, 1/2-inch wide and about 1/4-inch deep. The transverse cut gives a higher amplitude deflection than the longitudinal cut demonstrating that flaw indication is dependent on the shape and orientation of the defect as well as depth. There is also a certain amount of minor variation in the record which is particularly noticeable in the section of pipe which has the flaws in it. This is considered a background noise. It is believed due to variations in electrical resistivity and magnetic permeability. As seen in equation (1) the signal in the pick-up coil is influenced by resistivity, permeability and frequency as well as the desired variable, thickness (d). Frequency, of course, is held constant, but resistivity and permeability can vary. These variations may be due to heterogeneity in composition, heat treatment, rolling strains, straightening, etc. The noise level shown in this and following records is typical. It seldom amounts to more than a few percent equivalent thickness variation al-

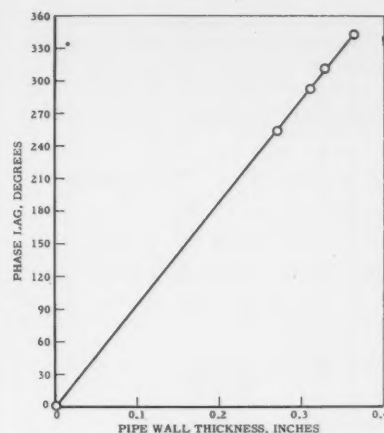


Figure 5—Phase lag of pickup signal with respect to exciter as a function of wall thickness (7-inch 26-pound J55 casing, 5 3/4-inch coils, 16 inch spacing, 60 cycles per second).

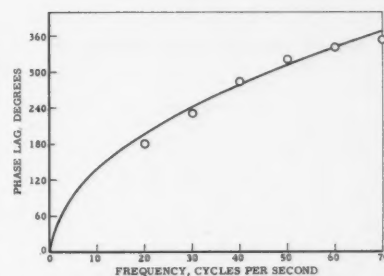


Figure 6—Phase lag of pickup signal with respect to exciter as a function of frequency (7-inch 26-pound J55 casing, 16-inch spacing, 0.362-inch wall thickness).

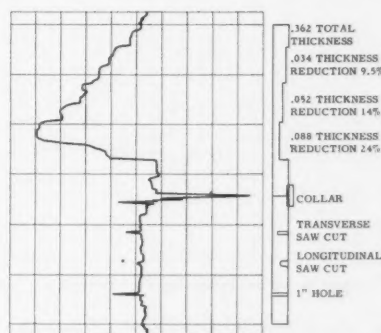


Figure 7—Seven-inch 26-pound J55 laboratory casing sample and typical log.

though occasionally unusually noisy or alternatively quiet pipe is encountered. This noise level does constitute a background level, however, over which a flaw indication must ride if it is to be recognized and thus is a limit to flaw sensitivity of the instrument. A representative figure for the limit of flaw sensitivity of the present instrument in average noise level pipe would be a local removal of metal equivalent to a drilled 1/2-inch hole in 7-inch casing. A redeeming feature of naturally occurring corrosion in oil wells is that it often occurs in fairly large patches, or in a scattering of pits some

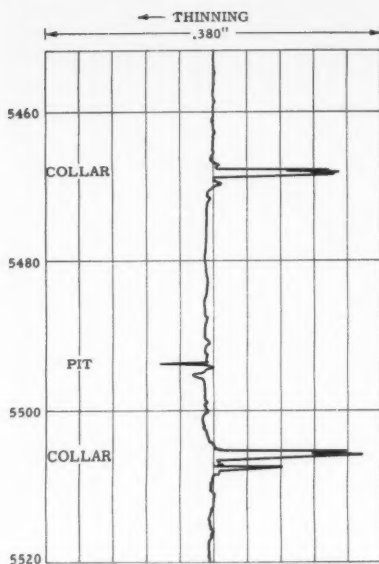


Figure 8—Section of well log illustrating pit.

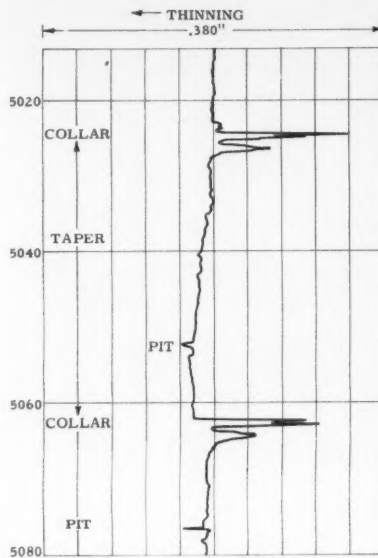


Figure 10—Section of well log illustrating taper and pits.

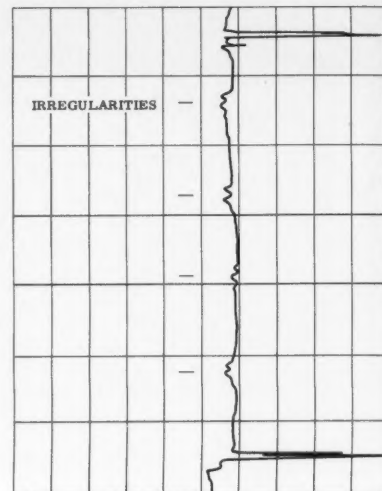


Figure 12—Section of well log illustrating manufacturing irregularities.

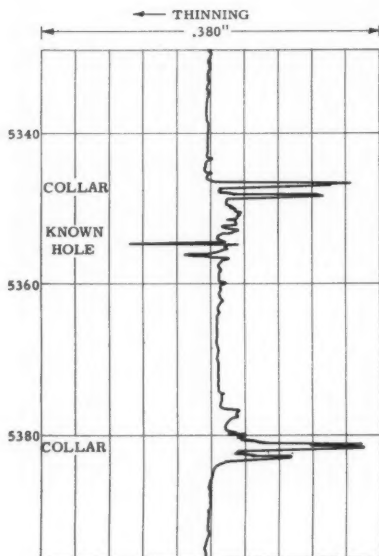


Figure 9—Section of well log illustrating hole.

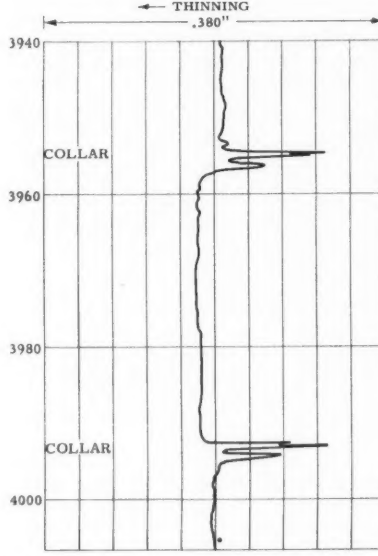


Figure 11—Section of well log illustrating general thinning.

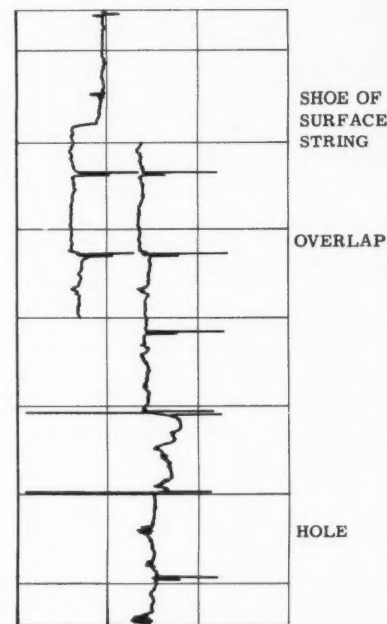


Figure 13—Section of well log illustrating shoe of surface string.

of which are large enough to demark the zone.

An actual pit occurring in an oil well is shown in Figure 8. Thinning is toward the left of the chart. The double break is quite evident with the sharper indication due to the pick-up. This indication was known to be a pit and not a hole in that the well was pressure tested just prior to logging and showed no leakage.

An actual known hole which had been repaired on two previous occasions is shown in Figure 9. Since this hole had been repaired by the usual squeeze cementing process, the absence of steel is still detected even though there was no leakage. The double indication is again quite evident, and the amplitude of deflection is about twice that of the previous pit.

While a certain amount of evidence of oil well corrosion indicates that corrosion occurs in isolated patches, the tool may be called upon to detect general corrosion thinning or drill pipe wear. Its ability to do this is demonstrated not only in Figure 7, the laboratory sample, but also in Figure 10 where a 0.020-inch taper is indicated in wall thickness and in Figure 11 where a section of casing 0.015-inch to 0.020-inch thinner than the rest is indicated. Figure 10 also shows evidence of two pits. The effect shown in Figure 11 of a piece of casing indicating a slightly different thickness than the piece above it or below it is quite common in wells. It is thought to be due to normal mill tolerances on these tubular goods and in part, differences in alloy, heat treatment, etc.

An interesting effect is shown in Figure 12. There were four equally spaced indications on over half the pieces of casing logged in this particular well. Because of the regularity of these indications, they are thought to be the result of some feature of the manufacturing process of this pipe which was known to have all come from the same manufacturer.

The surface string casing shoe is of interest. At this point the continuous oil string casing telescopes into the upper surface string and continues within it to the surface. It is a possible source of corrosion in that any electrical currents flowing up the oil string may jump over to the parallel path provided by the surface string. The point at which they leave

the oil string may be subject to corrosion damage.

Figure 13 shows this zone in an actual well. The shoe is located at 990 feet on the log. The record shows a stepwise thinning at this point, indicating a shielding effect of the surface string on the instrument but does not indicate any corrosion. The collar indications are also much changed in character. Because of a heavy attenuation of the pick-up signal within the surface string which makes logging and interpretation difficult, logs are usually not taken in this region. Corrosion pitting is indicated on the third piece of casing below the bottom of the surface string at a depth of 1083 feet. There are also numerous other indications of pitting in this area and a leak was subsequently detected by straddle packers in the interval 1219 to 1224. The log within this interval shows corrosion damage at 1221 feet. This particular log shows an example of reproducibility of

the instrument in that about 100 feet of pipe were relogged as a result of recentering the trace after emerging from the surface string.

Conclusions

The equipment has been in experimental use for about one and one-half years and a number of wells representing some 40,000 feet of pipe have been logged. These runs have been in the nature of field tests. There are several patentable features of the instrument* and at present there are two licensees. They are working on commercial versions of the instrument which should soon be available on a service basis.

While interpretation of the logs seems fairly straightforward in that the scale of average thickness is linear and that the instrument has been so designed that thinnings such as pits indicate only in

*For example, see MacLean W. R., U. S. Patent 2,573,799.

the thinning direction, there are occasionally unexplained features on the logs which will require additional experience to understand. Experience will also determine if the frequency of high background noise pipe will limit the usefulness of the instrument, although initial experience is encouraging. In the few pieces of noisy pipe encountered to date the noise had a regular or cyclic pattern which would enable pits or holes to still be distinguishable.

Through the use of the instrument previously unobtainable, information can be gathered on casing corrosion or casing deterioration in general, which should ultimately lead to a better understanding of its causes and its cures.

References

1. G. P. Harnwell. Principles of Electricity and Electromagnetism (First edition), McGraw-Hill Book Company, New York, (1936), p. 313.
2. F. E. Terman. Electronic and Radio Engineering (Fourth edition), McGraw-Hill Book Company, New York, (1955), p. 23.

Any discussion of this article not published above
will appear in December, 1961 issue.

Influence of Cold-Reduction and Heat-Treatment Combinations On Hydrogen Solubility and Permeability in Steel*

By R. M. HUDSON, K. J. RIEDY and G. L. STRAGAND

Introduction

IN EARLIER work reported in this journal,^{1,2} the effect of increasing cold work was shown to increase the hydrogen solubility in steel as measured after pickling of the steel in acid. This finding confirmed the results reported by other observers.^{3,4} A maximum value of hydrogen-desorption rate in water and in dry nitrogen at 38 C occurred below 30 percent reduction. Severely cold-worked steel displayed a considerably lower desorption rate than less cold-worked steel or annealed materials, regardless of whether the environment was water or dried nitrogen. The importance of steel-surface reactions in the desorption of hydrogen from steel has been established⁵ and is the subject of continuing investigations.

The present study, concerning the influence of cold-reduction and heat-treatment combinations on hydrogen absorption by steel immersed in acid, was undertaken because of the practical importance of better understanding the behavior of hydrogen in steel.

Discussion of Experiment

Materials

To study the effect of cold reduction on hydrogen absorption by steel during acid pickling, sheet-steel specimens representing reductions up to 78 percent were prepared at the Applied Research Laboratory, United States Steel Corporation. From a hot-rolled slab of a mild steel of thickness 0.086 inch, a set of specimens was warm rolled at about 700 C to various thicknesses down to 0.0189 inch. The chemical composition of this steel is given in Table 1. All hot-rolled pieces were then given a light sandblasting treatment to remove scale and were then heat-treated for one hour at 650 C in a dry atmosphere containing 5 percent hydrogen and 95 percent nitrogen. All the pieces were then cold-reduced to a final thickness in the range 0.0074 to 0.0220 inch, the reductions ranging from 2.1 to 78.6 percent.

One set of the prepared specimens was examined in the as-cold-reduced condition (these results were previously reported in Reference 1, Table 4). Another set, having initial reductions of 2.1, 28.3, 47.0, 67.6, and 78.0 percent, was box-annealed for 4 hours at 650 C in a dry atmosphere containing 5 percent hydrogen and 95 percent nitrogen. A third group, having initial reductions of 3.2, 28.3, 45.2, 66.8, and 78.1 percent, was box-annealed for 4 hours at 650 C in a dry atmosphere containing 5 percent hydrogen and 95 percent nitrogen, and all

Abstract

Tests were made to determine the effect of cold reduction on hydrogen solubility in steel. It was found that this direct relationship persisted through subsequent heat-treatment and later cold reduction. This was not true, though, of hydrogen permeability. Increasing the amount of cold reduction increased the hydrogen solubility. Increasing the amount of cold reduction also increased the hydrogen permeability until a maximum was reached (below 30 percent reduction for initially hot-rolled steel), after which the permeability decreased as the amount of cold work increased. 3.7.3

the specimens from this group were then given second cold reductions of 60 percent. A fourth group, having an initial cold reduction of about 78 percent, was box-annealed for 4 hours at 650 C in a dry atmosphere containing 5 percent hydrogen and 95 percent nitrogen, and specimens from this group were then given second cold reductions that varied from 0 to 60 percent.

All the pieces were sheared into 2- by 3/8-inch specimens, which were vapor-degreased with trichloroethylene prior to use.

Apparatus and Procedures

Specimens to be charged with hydrogen were immersed in 2N sulfuric acid solutions at 38 C for various times, after which they were removed from the acid solution and thoroughly rinsed with tap water for 15 seconds. The specimens were then given a final rinse with distilled water for 5 seconds before determination of hydrogen. The hydrogen concentration in the charged steel was determined by the warm-extraction method.⁶ In this method, specimens were placed in specially constructed borosilicate-glass extractors and heated for 16 hours at 160 C under mercury. The hydrogen

extracted was collected over mercury and measured volumetrically. The hydrogen concentrations given in this paper are in cubic centimeters (standard temperature and pressure) per 100 grams of steel.

Results and Discussion

For all the specimens examined, the hydrogen concentration increased linearly with the square root of the time that the specimen was immersed in acid until a saturation concentration was attained. This behavior is typical for hydrogen charging by acid pickling.^{1,2,3,5,6}

Although a detailed study of pickling rate was not made, some data on weight loss during pickling were obtained. Table 2 gives weight-loss information for 1-hour pickling in 2N sulfuric acid at 38 C. Multiplication of weight loss in mg by the factor 0.096 converts the data to grams per square foot of surface area. For the first group of specimens (those that were prepared by the cold reduction of hot-rolled strip), the increase in weight loss with increase in cold work demonstrates the marked effect that cold work has on the pickling rate of steel. The second group was prepared by annealing for 4 hours at 650 C a set of samples

TABLE 1—Chemical Composition of the Steel

Element	Composition, Percent
C	0.14
Mn	0.48
P	0.009
S	0.030
Si	0.013
Cu	0.02
Ni	0.01
Cr	0.02
N	0.010
As	0.008

TABLE 2—Pickling of Steel in 2N H₂SO₄ at 38 C as a Function of Cold Work

Initial Cold Reduction, Percent	Box Anneal (4 hrs at 650 C)	Second Cold Reduction, Percent	Finish Gage, Inch	Weight Loss, mg, after one-hour Pickling
3.7	No	No	0.0182	83
28.3	No	No	0.0190	85
47.3	No	No	0.0203	89
67.6	No	No	0.0194	99
78.2	No	No	0.0185	111
2.1	Yes	No	0.0189	41
28.3	Yes	No	0.0197	45
47.0	Yes	No	0.0220	51
67.6	Yes	No	0.0213	55
78.0	Yes	No	0.0201	51
3.2	Yes	60	0.0079	78
28.3	Yes	60	0.0082	76
45.2	Yes	60	0.0082	69
66.8	Yes	60	0.0083	73
78.1	Yes	60	0.0074	76
78.0	Yes	0	0.0201	51
78.3	Yes	15	0.0186	48
78.6	Yes	30	0.0132	55
78.0	Yes	45	0.0102	69
78.1	Yes	60	0.0074	76

* Submitted for publication July 18, 1960.

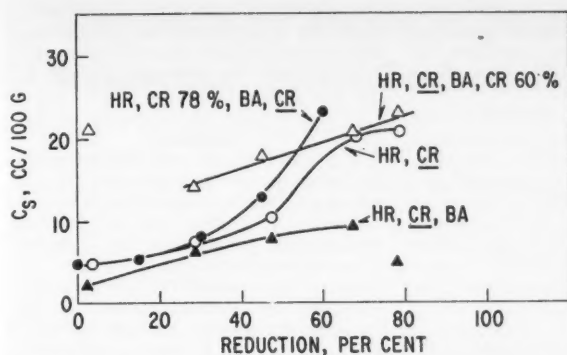


Figure 1—Effect of cold reductions and heat-treatment combinations on hydrogen solubility in steel.

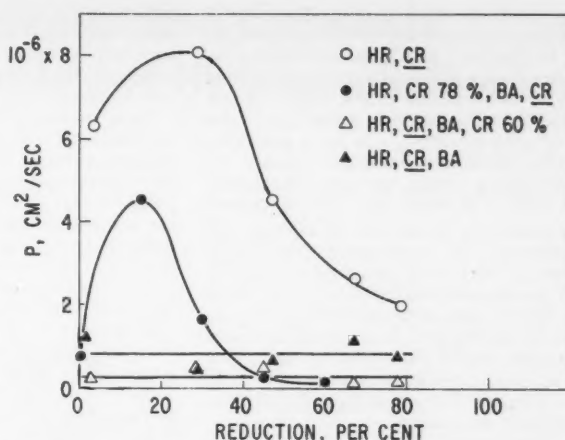


Figure 2—Effect of cold reduction and heat-treatment combinations on hydrogen permeability in steel.

TABLE 3—Effect of Cold-Work and Heat-Treatment Combinations on Hydrogen Solubility and Hydrogen Permeability in Steel

Initial Cold Reduction, Percent	Box Anneal (4 hrs at 650 C)	Second Cold Reduction, Percent	Hydrogen Solubility, cc/100g	Hydrogen Permeability,* P x 10 ⁶
3.7	No	No	4.7	6.3
28.3	No	No	7.1	8.1
47.3	No	No	10.2	4.5
67.6	No	No	20.0	2.6
78.2	No	No	20.9	2.0
2.1	Yes	No	2.1	1.2
28.3	Yes	No	6.2	0.43
47.0	Yes	No	7.3	0.61
67.6	Yes	No	9.0	1.1
78.0	Yes	No	4.8	0.75
3.2	Yes	60	21.2	0.19
28.3	Yes	60	14.2	0.44
45.2	Yes	60	17.9	0.46
66.8	Yes	60	20.2	0.09
78.1	Yes	60	23.0	0.09
78.0	Yes	0	4.8	0.75
78.3	Yes	15	5.2	4.5
78.6	Yes	30	7.7	1.6
78.0	Yes	45	12.8	0.18
78.1	Yes	60	23.0	0.09

* Unit for permeability is cm² per second.

Hudson



Riedy

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Stragand

having initial cold reductions similar to the first group. The weight losses observed for the annealed material did not show a dependence on the degree of original cold reduction employed. The third group had had various degrees of initial cold reduction, but all were sub-

sequently annealed for 4 hours at 650 C followed by a final cold reduction of 60 percent. The weight losses observed for this group did not show a dependence on the degree of original cold reduction employed. The last group was prepared by annealing the specimens for 4 hours at 650 C after an initial cold reduction of 78 percent and then giving them final reductions from 0 to 60 percent. The weight losses observed for this group generally increased with the increase in degree of final reduction.

In Table 3 and in Figures 1 and 2 are shown the change in hydrogen solubility, C_s , and hydrogen permeability, P , as a function of cold-reduction and heat-treatment combinations. The hydrogen-solubility values were taken from the saturation plateaus obtained on the hydrogen-absorption curves. The hydrogen permeability, P , is defined as the quantity of hydrogen (cc at standard temperature and pressure) transferred in 1 second through a diaphragm 1 millimeter thick and 1 square centimeter in cross section. The values given in this report were obtained by using the equation

$$P = 1.25 \rho \frac{\lambda^2 b^2}{C_s} \quad (1)$$

derived by Darken and Smith,³ where λ is the slope of the hydrogen-absorption curve (the hydrogen concentration in the steel plotted against the square root of time in seconds, that is, cubic centimeters per gram per (second)^{1/2}, C_s is the hydrogen solubility in cubic centimeters per gram, ρ is the density of the steel in grams per cubic centimeter, and b is the thickness of the specimen in millimeters. By using the data obtained in the present study and changing the units involved, hydrogen permeabilities in square centimeters per second were computed from the derived expression

$$P = 2.73 \times 10^{-7} \frac{\lambda^2 b^2}{C_s} \quad (2)$$

where λ is in cubic centimeters per 100 grams per (hour)^{1/2}, C_s in cubic centimeters per 100 grams, b in millimeters, and the density of steel is taken as 7.8 grams per cubic centimeter. The steel thicknesses used in these computations were estimated from weight-loss data for

one half the time required to reach a hydrogen-saturation level for each sample.

In Figures 1 and 2, the hydrogen solubilities and permeabilities are plotted either as a function of the initial amount of cold reduction or as a function of the final cold reduction, and for a particular curve, these functions are denoted by underlining. Thus, the curve designated as "HR, CR 78%, BA, CR" is plotted as a function of the amount of final cold reduction; the materials in this group were all hot-rolled, cold-reduced initially 78 percent and box-annealed for 4 hours at 650 C and then cold-reduced 0, 15, 30, 45, and 60 percent. It should be noted that for two of the solubility curves, one point is not consistent with the others in each group. The reasons for these deviations are not known.

From inspection of Table 3 and Figures 1 and 2, the following conclusions may be drawn: Cold reduction of hot-rolled strip increased the hydrogen solubility markedly, as was reported previously.¹ Annealing the cold-reduced strip lowered the hydrogen solubility from that of the cold-reduced condition, but the effect of the initial cold reduction persisted, the highest solubilities for the as-annealed specimens being related to the highest initial reductions. When cold-reduced and annealed steel was given a second cold reduction of 60 percent, the highest solubilities were found for steel having the

highest initial reductions. For steel initially cold-reduced 78 percent and then annealed, the hydrogen solubility increased as the amount of final cold reduction increased.

Cold reduction of the hot-rolled strip increased the hydrogen permeability to a maximum below 30 percent reduction, and further cold reduction lowered the permeability. This finding is similar to the results of Schumann and Erdmann-Jesnitzer,⁷ who reported that the rate of passage of electrolytically charged hydrogen through low-carbon steel diaphragms increased with an increasing amount of cold work up to 10 to 20 percent reduction, and that this rate decreased rapidly with further cold work. Annealing the cold-reduced strip lowered the hydrogen permeability from that of the cold-reduced condition to an essentially constant value, and unlike the hydrogen-solubility change, the effect of the initial cold reduction did not persist. When cold-reduced and annealed steel was given a second cold reduction of 60 percent, the resulting hydrogen permeability was nearly constant, regardless of the amount of initial cold reduction, and was at a low level compared with the other conditions examined. For steel initially cold-reduced 78 percent and then annealed, the hydrogen permeability increased with an increasing amount of final cold reduction to a maximum (probably below 20 percent reduction) and

then decreased with further cold reduction; the magnitude of permeability values for specimens in this group was less than that for the values found for the first group.

Summary

The effect of cold reduction on hydrogen solubility in steel persisted through subsequent heat-treatment and later cold reduction. This effect was not observed on hydrogen permeability, as computed from hydrogen-absorption curves. Increasing the amount of cold reduction increased the hydrogen solubility. Increasing the amount of cold reduction also increased the hydrogen permeability until a maximum was reached (below 30 percent reduction for initially hot-rolled steel), after which the permeability decreased as the amount of cold work increased.

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Corrosion of Steels in Boiling Potassium Carbonate Saturated With Carbon Dioxide and Hydrogen Sulfide*

By D. BIENSTOCK and J. H. FIELD

Introduction

IN DEVELOPING the hot-carbonate process^{1,2} for scrubbing carbon dioxide and hydrogen sulfide from gas mixtures with a potassium carbonate solution, corrosion of the steel equipment occurred. The corrosion was especially severe in scrubbing carbon dioxide. Reboiler tubes were attacked and the erosion of pump impellers, valve seats and piping downstream from the pressure letdown valve occurred. Because no experimental corrosion data with this reaction mixture at 110 C were available, a laboratory investigation on rates of corrosion and use of inhibitors was started.

Experimental Procedure

Carbon-steel discs were cut from a cold-rolled sheet, 20 U.S. Standard gauge, and machined to a diameter of 2.232 inches. Earlier test with discs cut from hot-rolled sheets gave no significantly different results. The discs were polished with 240-grit carborundum paper, washed, dried, and weighed. Thickness was measured with a micrometer. The discs were mounted on a spool-type holder³ of bakelite and separated by 7/16-inch bakelite spacers.

A photograph of the assembly is shown in Figure 1. The corrosion vessel is of stainless steel, 8 inches in diameter x 9 3/4 inches in height. The spool was immersed in 4 liters of test solution and the system was flushed with helium to purge it of oxygen. When an acid gas was to be used in the test, 0.4 cubic feet per hour of carbon dioxide or a carbon dioxide mixture with hydrogen sulfide was admitted, and the solution was brought to boiling.

At the end of the test the discs were removed, washed with water, and scrubbed with 3/0 steel wool. The cleaned discs were dipped in acetone and dried in air. The cleaning was repeated until the difference in weight between two successive cleanings was less than 1 milligram. Figures 2 and 3 show the corrosion unit and a schematic flow diagram, respectively. Tests were made simultaneously in four corrosion vessels. Because of the extended periods of operation, the corrosion-testing equipment was designed to require little attention by plant personnel. Some operational aspects were as follows:

(1) A constant head device was used for controlling the flow of carbon dioxide and hydrogen sulfide. Although more gas was thus consumed, only occasional throttling was required.

(2) Purge lines vented to the outside



Field

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were heated by steam coils to prevent plugging in freezing weather.

(3) Calibrated, sensitive thermometers were used to indicate changes in concentration of solution. Loss of water from a steam leak or by volatilization in the gas would change the temperature of the boiling solution.

(4) Because a perforated or fritted disc to disperse the feed gas in the solution would eventually plug with caked solid, an expanded open end tube was substituted. As the corroding liquid was boiling, it was felt that adequate dispersion of the gas was obtained.

(5) Corrosion discs once used in a test, even those showing minor attack, were not reused in subsequent tests.

(6) The carbon dioxide contained less than 0.05 volume percent of oxygen.

(7) The Tutwiler method⁴ was used in determining the concentration of hydrogen sulfide.

The following corrosion tests were performed:

1. In K_2CO_3 solutions.

Abstract

Corrosion rates of carbon and stainless steels were determined in boiling solutions of potassium carbonate saturated with carbon dioxide and hydrogen sulfide. Solutions saturated with carbon dioxide were highly corrosive to carbon steel and slightly corrosive to Type 304 and 347 stainless. Type 410 stainless behaved similarly to carbon steel. Monel was even more resistant to attack. The addition of only 0.3 percent of hydrogen sulfide to the carbon dioxide reduced corrosion rate of the carbon steel by 96 percent. Boiling solutions of carbonate saturated with hydrogen sulfide were noncorrosive. 4.3.6

2. In the $K_2CO_3-CO_2$ system.
3. In the $K_2CO_3-CO_2-H_2S$ system.
4. In the $K_2CO_3-H_2S$ system.

Discussion

Corrosion in Potassium Carbonate Solutions

The rate of corrosion of mild carbon steel in boiling solutions of 30 to 45 percent potassium carbonate was determined. The results are shown in Table 1. The corrosion rates vary from 10 to 20 mils per year. X-ray analysis of the corrosion products on the metal surface indicated the presence of Fe_2O_3 .

Corrosion in Potassium Carbonate-Carbon Dioxide System

As shown in Table 1 and plotted in Figure 4, the rate of corrosion increased markedly in the presence of carbon dioxide. A 40-percent solution of potassium carbonate at 109 C in the absence of carbon dioxide has a corrosion rate of 11 mils per year; in the presence of carbon

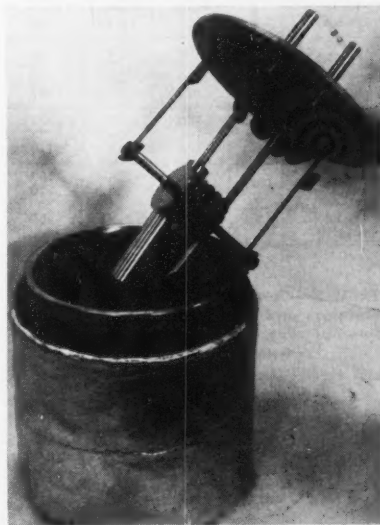


Figure 1—Spool-type holder for corrosion discs.

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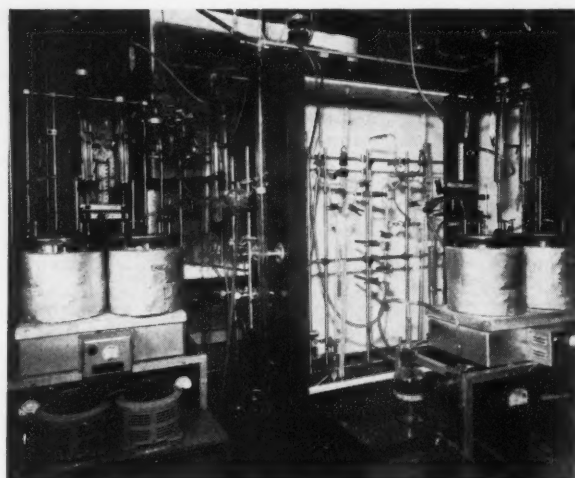


Figure 2—Laboratory corrosion testing apparatus.

dioxide the rate increased to 340. Under the conditions of the laboratory test, in which carbon dioxide is bubbled into the boiling carbonate solution, 15 to 20 percent conversion of the carbonate to the bicarbonate occurs. This conversion corresponds approximately to a two-stage, split-stream regeneration in a commercial unit. The corrosion of the steel, shown in Figure 5, results in a uniform reduction of the metal; pitting is rarely observed.

Two austenitic stainless steels (Types 304 and 347), a martensitic stainless (Type 410), and Monel were also tested; a boiling 40-percent carbonate solution saturated with carbon dioxide was used. As shown in Table 2 the austenitic steels were slightly attacked (1 mil per year), and the Monel even less (0.1 mil per

year). The martensitic steel had the same corrosion rate as the carbon steel—300 mils per year.

Corrosion in Potassium Carbonate-Carbon Dioxide-Hydrogen Sulfide System

Mixtures of carbon dioxide and hydrogen sulfide containing 0.3 to 10 percent hydrogen sulfide were bubbled into boiling solutions of 40 percent potassium carbonate. This range in concentration is approached industrially in the scrubbing of mixtures containing these gases. As shown in Table 3 the rates of corrosion were all approximately 10 mils per year. Thus the addition of only 0.3 percent hydrogen sulfide to carbon dioxide decreases the corrosion rate by 96 percent. Scrubbing of a gas containing carbon dioxide as the only acidic constituent with a hot noninhibited solution of potassium carbonate could not be done in mild steel equipment because of the excessive corrosion that would occur. However, if the gas contains a small amount of hydrogen sulfide, the reduced rate of corrosion could then be tolerated.

Increasing the concentration of a boiling solution of carbonate saturated with carbon dioxide and hydrogen sulfide results in an increase in corrosion. As shown in Table 4, the corrosion rate increased from 8 to 40 mils per year as the carbonate concentration was increased from 20 to 50 percent. In the commercial application of hot carbonate solutions for scrubbing acid gases, the concentration of potassium carbonate varies from 25 to 33 percent.

TABLE 1—Corrosion of Carbon Steel in Boiling Solutions of Potassium Carbonate Alone and Saturated With Carbon Dioxide

Concentration of K_2CO_3 , Weight Percent	Gas	Temperature, °C.	Length of Test, Days	Corrosion Rate, mpy
30.....	None	104	28	14
35.....	None	106	25	13
40.....	None	109	28	11
45.....	None	111	27	17
0.....	CO_2	100	15	1
15.....	CO_2	101	14	33
25.....	CO_2	103	14	110
30.....	CO_2	104	14	160
35.....	CO_2	106	14	270
40.....	CO_2	109	14	340
50.....	CO_2	115	12	460

TABLE 2—Corrosion Rates of Carbon Steel, Stainless and Monel in a 40-percent Solution of Potassium Carbonate Saturated with Carbon Dioxide*

METAL	Length of Test, Days	Corrosion Rate, mpy
Carbon steel.....	14	340
AISI Type 304 stainless steel.....	29	1.0
AISI Type 347 stainless steel.....	29	0.7
AISI Type 410 stainless steel.....	14	310
Monel.....	21	0.1

* Temperature of boiling solution: 109C

TABLE 3—Corrosion Rates of Carbon Steel in a 40-percent Solution of Potassium Carbonate Saturated with Mixtures of Carbon Dioxide and Hydrogen Sulfide*

$CO_2:H_2S$ Ratio	Length of Test, Days	Corrosion Rate, mpy
90:10.....	29	10
97:3.....	30	8
99:1.....	30	16
99.7:0.3.....	30	12

* Temperature of boiling solution: 109C

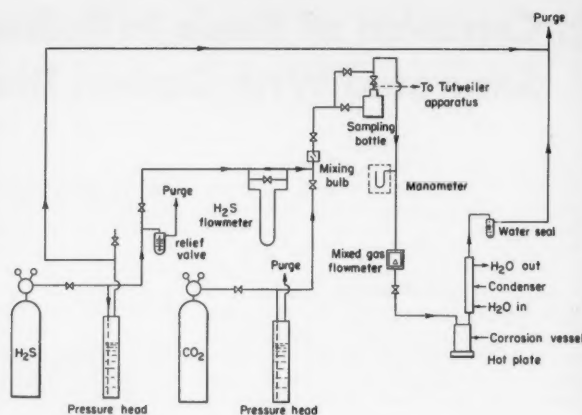


Figure 3—Schematic flow diagram of corrosion testing apparatus.

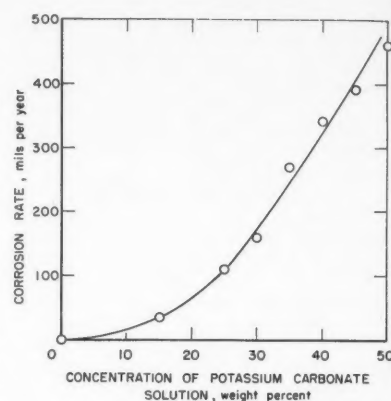


Figure 4—Corrosion rates of carbon steel in boiling solutions of potassium carbonate saturated with carbon dioxide.

Lowering the temperature, while maintaining the same concentration of carbonate, reduced the corrosion rate. As shown in Table 5, with a 30 percent solution of carbonate saturated with carbon dioxide and hydrogen sulfide, the corrosion rate was reduced 90 percent in lowering the temperature from 104 to 90 C. A 90 percent reduction in corrosion was also observed when the temperature was lowered to 90 C in a 30 percent solution of carbonate saturated with carbon dioxide. Concentrations of potassium carbonate above 30 percent could not be maintained at 90 C owing to precipitation of solids. Temperatures of 90 C (194 F) are used in split-stream operation for reducing the carbon dioxide content to 1 percent and lower (a 2-percent concentration is usually obtained with the conventional operation). In the split-stream

TABLE 4—Corrosion Rates of Carbon Steel in Boiling Solutions of Potassium Carbonate*

Concentration of K_2CO_3 , Weight Percent	Temperature, °C	Length of Test, Days	Corrosion Rate, mpy
30.....	104	30	8
40.....	109	30	12
50.....	115	30	40

* Saturated with a mixture of carbon dioxide and hydrogen sulfide in a ratio of 99.7 to 0.3

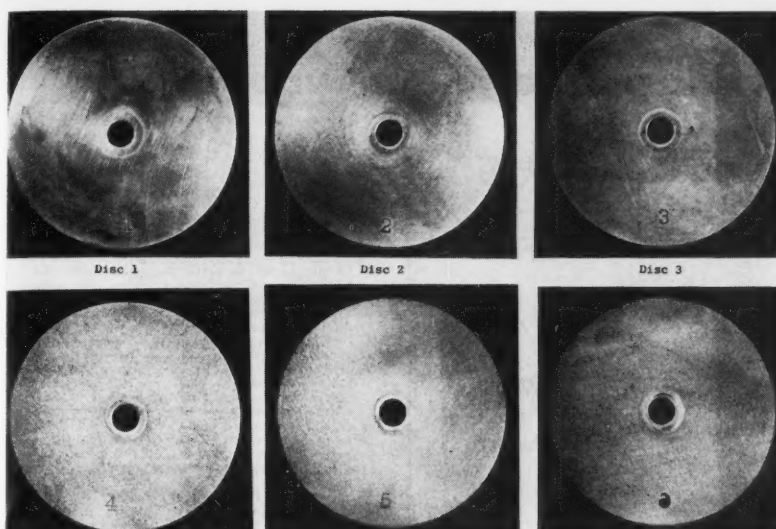


Figure 5—Corrosion of carbon steel in a boiling 50 percent solution of potassium carbonate saturated with carbon dioxide. Disc 1—one day; Disc 2—two days; Disc 3—three days; Disc 4—six days; Disc 5—nine days; Disc 6—twelve days.

method about one-third of the solution leaving the regenerator is cooled to 90 C and pumped to the top of the absorber.

The rates of corrosion of several stainless steels and wrought iron are compared with carbon steel in Table 6. The 18-8 chromium-nickel austenitic stainless steels (304, 347) have negligible corrosion in the system K_2CO_3 - CO_2 - H_2S and such steels can be used in areas where corrosion difficulties are more likely to occur. The 410 martensitic and the wrought iron have the same corrosion rate as carbon steel.

Corrosion in Potassium Carbonate-Hydrogen Sulfide System

Because hydrogen sulfide so effectively reduces the corrosion when mixed with carbon dioxide in a carbonate solution, a test was made to ascertain if hydrogen sulfide alone is corrosive. It was found that in a boiling solution of 40 percent potassium carbonate saturated with hydrogen sulfide, complete inhibition of corrosion was obtained. A bluish-black film formed which was not removed by polishing with steel wool. The film re-

sulted in a small weight increase of the discs.

Inhibitors

Use of inhibitors in the hot carbonate system is under investigation. Numerous reagents have been tested, and a report of these findings will be published in the near future.

Conclusions

Solutions of potassium carbonate used in absorbing carbon dioxide and hydrogen sulfide are corrosive to steel equipment. A laboratory corrosion study has shown that boiling solutions of potassium carbonate saturated with carbon dioxide corroded carbon steel at the rate of 340 mils per year. The 300 series of stainless steels such as 304 and 347 suffered negligible attack in this mixture (the corrosion rate was about 1 mil). Monel was even more resistant to attack; its corrosion rate was 0.1 mil. The 410-stainless steel behaved similarly to carbon steel.

Any discussion of this article not published above will appear in December, 1961 issue.

TABLE 5—Effect of Temperature on Corrosion Rate of Carbon Steel in a 30 Percent Solution of Potassium Carbonate*

GAS	Temperature, °C	Length of Test, Days	Corrosion Rate, mpy
$CO_2 + H_2S$	104 (boiling)	14	8
$CO_2 + H_2S$	100	14	7
$CO_2 + H_2S$	90	14	0.7
CO_2	104 (boiling)	14	158
CO_2	90	14	17

* Saturated with a mixture of carbon dioxide and hydrogen sulfide in a ratio of 97 to 3 and with carbon dioxide alone.

TABLE 6—Corrosion in a 40 Percent Solution of Potassium Carbonate Saturated with Carbon Dioxide and Hydrogen Sulfide*

METAL	$CO_2:H_2S$ Ratio	Length of Test, Days	Corrosion Rate, mpy
Carbon steel....	97:3	30	8
304 stainless....	97:3	30	0.03
347 stainless....	97:3	30	.05
410 stainless....	97:3	30	9
Carbon steel....	99.7:0.3	30	12
Wrought iron....	99.7:0.3	30	8

* Temperature of boiling solution: 109 C

When hydrogen sulfide was present with the carbon dioxide the corrosiveness of the solution was considerably reduced. A 40 percent solution of potassium carbonate saturated with carbon dioxide and hydrogen sulfide and containing from 0.3 to 10 percent hydrogen sulfide corroded carbon steel at the rate of 10 mils per year—a reduction of 96 percent. This lower rate of corrosion can be tolerated in carbon steel equipment of satisfactory design.

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Stress-Corrosion Cracking: A Review of Current Status*

By W. LEE WILLIAMS

Introduction

STRESS-CORROSION cracking is a term that lacks a universally accepted definition. It might be well at the outset to discuss the meaning of this and certain related terms. Only in this way is it possible to gain understanding of the subject matter covered in this lecture.

Stress Corrosion and Stress-Corrosion Cracking

First, it should be recognized that common usage makes a distinction between stress corrosion and stress-corrosion cracking. The Corrosion Handbook¹ defines these terms as follows:

(a) Stress corrosion is any combined effect of stress and corrosion on the behavior of metals.

(b) Stress-corrosion cracking is the combined action of static stress and corrosion which leads to cracking or embrittlement of metals.

There are at least two reasons why the latter term is defined separately. The first reason is that stress can have an effect on general corrosion attack without local cracking or embrittlement. This can be seen in Figures 1a and 1b, in which the stressed section shows the greater degree of general wastage, other factors being equal. Examples of acceleration of general corrosion have been cited in the literature (e.g., Uhlig¹ and Evans²). The effects are usually small and tend to be overshadowed by other variables. This is not particularly surprising, as the effect of elastic stress on potential is very small. Yang, Horne and Pound³ have indicated that the observed magnitude of potential shift lies somewhere between 0.01 and 0.1 mv per 1000 psi applied elastic stress.

Figure 1c illustrates the case where the application of stress has not increased general corrosion wastage, but has been responsible for the generation of a crack which would not have formed if either the stress or the corrosive environment were absent. This is clearly the special case of stress corrosion known as stress-corrosion cracking.

The progression of the crack in this case may proceed by a combined corrosion and mechanical process, in which the mechanical aspect is of a type not present in the case of stress-accelerated general corrosion. This is the second reason why stress-corrosion cracking is differentiated from the more generalized term, stress corrosion. More will be said later about the mechanism of crack propagation.

Effects of corrosion are sometimes evaluated in terms of reduction in strength of a corroded part or specimen



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through loss of effective cross section. To illustrate this, Figures 1b and 1c were purposely drawn with equal residual cross section and reduction of strength. One might assume that, on the basis of the strength test, there could be a twilight zone in which it would be difficult to distinguish between stress-corrosion cracking and acceleration of corrosion by stress. Actually, such a circumstance would rarely, if ever, be encountered in actual practice.

In the first place, stress usually has no more than a minor effect on the rate of general corrosion attack. In the second place, stress-corrosion cracking which occurs in service is nearly always associated with only mild general corrosion. Thus, a more realistic representation of what one might expect to find is shown in Figure 2. As can be seen, there is usually little need to be concerned about the presence of stress unless the circumstances are likely to lead to stress-corrosion cracking, Figure 2c.

Static and Cyclic Stresses

Taking a closer look at the Corrosion Handbook's definition of stress-corrosion cracking, it is seen that the definition is specific in the use of static stress in lieu of stress. This is done purposely so that stress-corrosion cracking will not be confused with corrosion-fatigue cracking. It has been established that there are at least four differences which justify the distinction between cracking by stress corrosion and corrosion fatigue.

Abstract

Despite long interest and effort in the study of stress-corrosion cracking, the term itself still lacks a universally recognized definition. The paper discusses various definitions which have been used in the past. For the most part, these have been so generalized that they fail to distinguish among several modes of cracking. An attempt is made to clear up this apparent confusion by classifying the various modes of cracking and by illustrating each with typical examples. Topics discussed include static and cyclic stresses, tensile and compressive stresses, electrochemical mechanisms, molten phase attack, and hydrogen cracking.

Probably the most important type of stress-corrosion cracking, from a practical standpoint, is that which is now recognized as being associated with electrochemical phenomena. Many examples have been observed and studied, but only limited progress has been made toward a basic understanding of how the cracking initiates and progresses. This type of cracking involves a complex interplay of metallurgical and chemical factors, the study of which has taxed the ingenuity of experimenters. Definite progress has been made, however, and recent accomplishments are reviewed.

35.8

First, the cyclic stresses associated with the corrosion fatigue process may lead to the formation of cracks that would not form in some environments under conditions of static stress. Second, both stress-corrosion cracks and corrosion-fatigue cracks are commonly observed to be preceded by the formation of corrosion pits from which the cracks subsequently propagate. But McAdam⁴ has pointed out that the pits tend toward a saucer shape under stressless and static-stress conditions, whereas the pits tend toward greater depth and sharpness under cyclic-stress conditions. Third, McAdam⁵ has also pointed out that once these sharp pits have formed, the metal may have already been damaged to the extent that the cracks may continue to propagate at cyclic stresses below the normal endurance limit of the material, even if the corrosive environment be removed. In other words, the corrosion aspect acts as a means of producing initial stress concentration, the magnitude of which may be sufficient to complete the development of the crack by purely mechanical means.

Finally, it has been observed that corrosion-fatigue cracks are predominantly transgranular, even in metals which would normally fail intergranularly under conditions of stress-corrosion cracking. Knowledge of this is sometimes helpful in diagnosing the cause of service failures.

There are alloys which do fail by stress-corrosion cracking in a transgranular fashion. The stabilized austenitic stainless steels are an example. In such cases it is often difficult to tell the difference between corrosion-fatigue and stress-corrosion cracks by metallographic examination. Figure 3 shows a relatively

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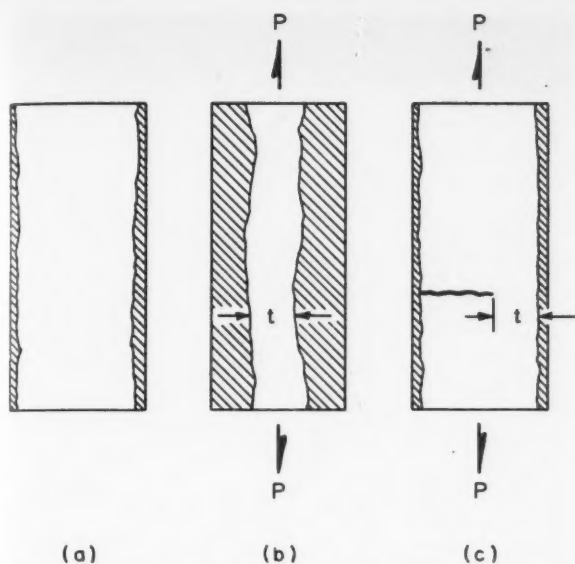


Figure 1—A possible representation of (a) general corrosion without stress, (b) acceleration of general corrosion by stress, and (c) stress-corrosion cracking.

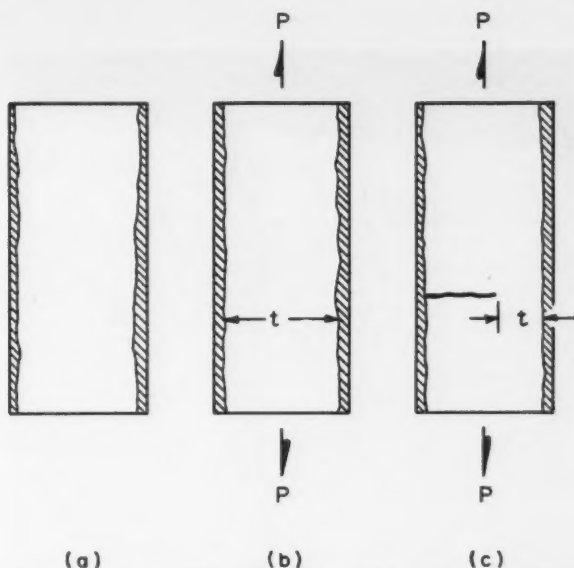


Figure 2—A more realistic representation of (a) general corrosion without stress, (b) general corrosion in the presence of stress, and (c) stress-corrosion cracking.

straight transgranular stress-corrosion crack in an austenitic stainless steel. A fatigue crack formed under conditions of cyclic uniaxial stress could have a similar appearance.

Figure 4 shows a highly branched network of stress-corrosion cracks in a similar steel. This network is certainly different from the usual fatigue crack. However, thermal fatigue cracks sometimes are branched like this. Thus, diagnosis of a service failure in a metal, particularly one which is subject to transgranular cracks from both stress corrosion and fatigue, requires knowledge of the circumstances leading to failure in addition to the usual metallographic evidence.

To recapitulate: Table 1, borrowed from a recent paper by Copson⁶, is a classification of various forms of stress corrosion. The approach and analysis used here closely follow that of Copson. However, Copson has provided for stress-accelerated pitting and other forms of attack in addition to general attack and cracking.

Tensile and Compressive Stresses

Note that Copson's definition of stress-corrosion cracking limits the stress to tensile stress. This is acceptable, as no case of stress-corrosion cracking has been experienced with compressive stresses. Indeed, the intentional introduction of compressive stresses, such as by peening, is one of the means sometimes used to combat stress-corrosion cracking.

There have been cases reported (e.g., Evans²), in which compressive stresses have accelerated corrosion, but cracking was not involved. This adds further support to the idea that stress-corrosion cracking is a special case wherein the progression of the crack is intimately associated with both corrosive and mechanical factors.

Types of Stress-Corrosion Cracking

Stress-corrosion cracking involves the combined action of stress and corrosion. But there is still some question about what kind of corrosion is being referred to. Should the word be interpreted in its broadest sense? If so, it must be understood that an explanation of the basic mechanism involved in one case may not apply to the next. Failure to recognize that several different processes may be involved has led to much confusion.

Several investigators have attempted to classify the various types of stress-corrosion cracking. The classification system discussed here is the one reproduced in Table 2, with slight editing, from a recent paper by Copson.⁶

Electrochemical Mechanisms

Heading the list is the type of stress-corrosion cracking associated with electrochemical action. This type probably represents the greatest interest from the standpoint of industrial importance. It is also the least understood, and the most difficult to predict and control.

Electrochemical action requires cathodic and anodic areas in the presence of an electrolyte. In the special case of stress-corrosion cracking, Dix⁷ proposed that there must exist a susceptibility to selective corrosion along more or less continuous anodic paths. Copson's table proposes four possible situations which can account for the existence or formation of such paths of sensitivity.

The first situation, A-1, is that in which an alloy contains a network of anodic constituents. Mears, Brown and Dix⁸ studied an example of this in aluminum-4 percent copper alloys. Suitable heat treatment caused precipitation of CuAl₂ at the grain boundaries, thereby depleting the alloy of copper in a network along the grain boundaries. The depleted network was anodic to both the CuAl₂ and the remaining matrix.

In some cases stress is not necessary for corrosion to occur along anodic networks, although the presence of stress will usually cause marked acceleration of attack. In other cases the presence of stress is required if attack is to occur. Dix⁷ reasoned that the principal function of the stress in such cases was to cause rupture of the protective surface film, thereby exposing fresh anodic material to the corrosive environment.

When austenitic stainless steels stress corrode, they usually do so along a predominantly transgranular path. However, if the stainless steel is not one of the stabilized grades, the path of failure can be shifted to the grain boundaries by a sensitization heat treatment. Williams and Eckel⁹ cited an example in the case of Type 304 stainless steel submerged in an alkaline-phosphate treated boiler water at 500 F. Nonsensitized steel did not stress corrode. After sensitization at 1200 F, the alloy cracked rapidly at the grain boundaries where the formation of carbides had produced a network depleted in chromium. Stress was required to produce attack in this case, as unstressed sensitized specimens showed no significant grain boundary corrosion.

Returning again to Table 2, the second situation, A-2, provides a crack-sensitive path by stress-accelerated decomposition. Unlike the first situation, which involved a pre-existing anodic path in the alloy, the second situation is thought to involve a path that is capable of self-generation during the stress-corrosion process.

It may be reasoned that the application of stress can cause local rupture of a protective film. The unprotected metal at the film rupture is anodic to the surrounding film, and local corrosion of the anode begins. This results in a pit which serves as a stress-concentrating notch. The high local strain at the notch then results in precipitation or formation



Figure 3—A relatively straight transgranular stress-corrosion crack in an austenitic stainless steel. Magnification 100X.

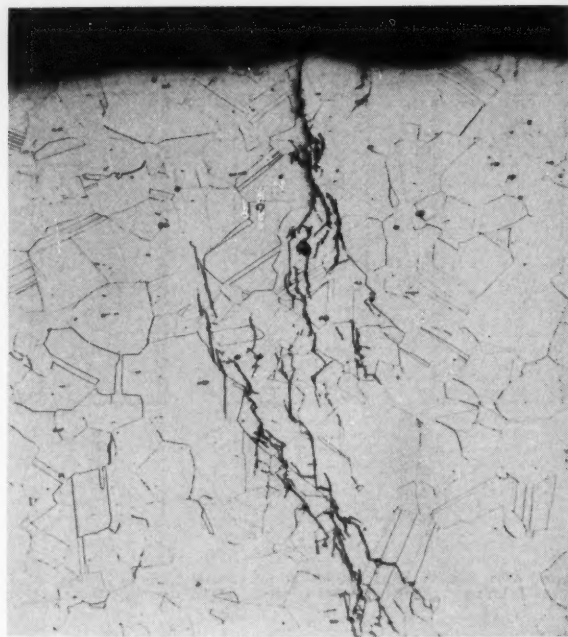


Figure 4—A branched network of transgranular stress-corrosion cracks in an austenitic stainless steel. Magnification 100X.

of a new phase which is anodic to the matrix. Corrosion at this anode site causes further stress concentration and repetition of the cycle.

Edeleanu¹⁰ suggested that the above mechanism occurred during transgranular stress-corrosion cracking of austenitic steel. He based his suggestion on the observation that cracks followed martensite plates formed by strain-induced austenite decomposition. This may account for some cases, but certainly not all. Williams and Eckel,⁹ for example, have shown that austenitic steels with completely stabilized austenite can stress corrode in the absence of martensite.

Copson's item A-3 suggests that local plastic deformation may provide a third situation leading to generation of an anodic crack-sensitive path. This may appear at first glance to be similar to the second situation. However, a different concept was in mind. The third situation depends on the formation of anode sites from strain alone, rather than on a strain-induced precipitate or new phase. This concept appears to have been derived partly from the work of Hoar and West,¹¹ who showed that anodic polarization of an austenitic stainless steel wire undergoing strain was less than for a wire in a static condition. This observation indicated greater ease of metal dissolution as the lattice was subjected to plastic deformation. The observation also would account for crack generation rather than general attack, as high strain would continue to be concentrated at the tip of an advancing crack.

In discussing the above concept, Uhlig¹² raised doubts as to whether the higher energy of a strained lattice could actually account for the rapid anodic dissolution rates observed during real stress-corrosion reactions. Uhlig's reason-

ing led to the proposal that an increase in reaction rate on cold working of metals is caused by composition gradients produced within the metal by plastic deformation. Similarly, increased reactivity along crack-sensitive paths is caused predominantly by composition differences along such paths, and to a smaller or negligible extent by differences in energy of atoms displaced from their normal lattice positions.

Uhlig accounts for strain-induced composition differences by the increased rates of diffusion which become possible in the vicinity of slip clusters and dislocation arrays. The accumulation of nitrogen at strain-induced imperfection sites was cited as a factor leading to stress-corrosion cracking in austenitic stainless steels. This seemed to be confirmed by experiments¹³ which indicated that greatly increased resistance to cracking in the boiling magnesium chloride test could be obtained by lowering of the nitrogen in stainless steels to very low levels.

If Uhlig's theories are correct, there would be reason to delete item A-3 from Copson's list of situations leading to the formation of crack-sensitive paths. However, it would require extension of the second item, A-2, to include not only the formation of stress-induced new phases, but also to include stress-induced composition gradients in the vicinity of lattice imperfections.

Item A-4 in Copson's table concerns the stress-induced breakdown of protective films. The stress-corrosion mechanism is pictured as starting with stress causing rupture of protective surface films. Corrosion then begins at the exposed anode sites. Thus far the circumstances are not different from the other situations. However, in this case, it is

suggested that the stress has a different function in relation to the propagation of the crack. It is thought that the corrosion of the initial anode forms a notch, that protective films reform along the sides of the notch, and that the stress concentration at the notch tip causes continuous local film rupture. Thus, the notches grow into cracks.

TABLE 1—Classification of Various Forms of Stress Corrosion*

Stress Corrosion —Combined action of stress and corrosion	
A.	Uniform attack
B.	Pitting
C.	Cracking
1.	Corrosion fatigue: combined action of corrosion and cyclic stress.
2.	Stress-corrosion cracking: combined action of corrosion and static tensile stress.
D.	Other forms of attack.

* From Copson⁶.

TABLE 2—Classification of Various Types of Stress-Corrosion Cracking*

Type A: Attack involving an electrochemical mechanism.	
1.	Anodic constituent at grain boundaries or elsewhere.
2.	Stress-accelerated decomposition.
3.	Anodic paths from local plastic deformation.
4.	Breakdown of protective films.
Type B: Attack involving a molten phase	
1.	Liquid metal.
2.	Fused corrosion product.
3.	Molten industrial product.
Type C: Attack involving hydrogen	
1.	Hydrogen generated by corrosion.
2.	Hydrogen generated by a galvanic couple.
3.	Hydrogen generated by an impressed current.
Type D: Attack involving other factors	
1.	Wedging action of corrosion products.
2.	Failures which are largely mechanical.
3.	Stress-sorption cracking.

* From Copson⁶, with slight changes.

There are many other interesting aspects associated with the electrochemical mechanisms of stress-corrosion cracking. An example is the research done on film formation and rupture, such as described by Logan,¹⁴ Hoar and Hines,¹⁵ and others. However, time will not permit further discussion during this lecture.

To summarize what has been said about the electrochemical mechanisms of stress-corrosion cracking:

1. The mechanisms involve an intimate and complex interplay of tensile stress, corrosion, and crack-sensitive anodic paths through the alloy.

2. The crack-sensitive paths may pre-exist in the alloy, or they may be self-generated ahead of the propagating crack.

3. The factors determining the initiation and propagation of stress-corrosion cracks include stress level, alloy composition, alloy condition, notch sensitivity, film formation, film rupture, corrosive environment, and perhaps others.

4. The controlling factors probably vary from case to case, so that a completely satisfactory generalized theory may be difficult and perhaps impossible to obtain.

5. Despite the complexities, great strides have been made toward understanding the basic reactions in electrochemical stress-corrosion cracking, and understanding is the first step toward rational control.

Molten Phase Attack

No lecture on stress-corrosion cracking would be complete without mention of other types, which do not depend on electrochemical action. These types, which constitute the remainder of Table 2, will be discussed briefly.

The type of attack listed under item B is that caused by reaction with a molten phase—either a liquid metal, a fused corrosion product, or a molten industrial product. Typical examples can be cited.

The intergranular season cracking of stressed brass in mercury, such as described by Edmunds¹⁶, is the best-known example of liquid metal attack. Another, described by Page¹⁷, is high-temperature intergranular attack and embrittlement of stressed steel by zinc. The latter example is of considerable industrial importance, and the American Society for Testing Materials has a recommended practice¹⁸ to safeguard against embrittlement of cold-worked steel products during hot galvanizing.

There are numerous examples of attack from fused corrosion products and molten industrial products. These differ mainly in the source of the corrosive constituents. In the case of industrial products, such as fused salts, the engineer usually is aware of the nature of the product to be handled, and materials for his equipment can be selected accordingly.

The situation may not be so readily apparent in the case of fused corrosion products. These products arise through reaction of the alloy with the environment, or through deposition of products directly from the environment. When the products are fused, they

sometimes cause selective attack and embrittlement of stressed alloys. Particularly troublesome are cases of mixed constituents which form eutectics with depressed melting points, such as in sulfated-vanadium fuel-oil slags.

Copson⁶ described grain-boundary stress-corrosion cracking of nickel in sulfide environments above 1190 F. This temperature corresponds to the melting point of the nickel-nickel sulfide eutectic. Other examples of high temperature attack were described by Wilkes¹⁹ in his paper on turbo-supercharger alloys tested in the products of combustion of leaded gasoline.

Hydrogen Cracking

The type of attack listed under item C involves embrittlement and cracking of certain metals with hydrogen. Strictly speaking, this is not a true corrosion process, and perhaps it should not be included in a lecture on stress-corrosion cracking. However, hydrogen cracking does have the characteristic of being stress-dependent, and it is often associated indirectly with corrosion problems. So it is well to describe hydrogen cracking, if for no other reason than to eliminate confusion with true stress-corrosion cracking.

Hydrogen cracking occurs transgranularly and is found most frequently in ferrous materials at high hardness levels. The ferrous materials have a body-centered cubic structure in which hydrogen has a rapid diffusion rate but limited solubility. This situation is conducive to the accumulation of hydrogen at imperfections and dislocations. Presumably, crack propagation proceeds by purely mechanical means, abetted by the lower ductility and greater notch sensitivity of the alloys at the higher hardness levels.

Three sources of hydrogen are indicated in Table 2. The first source is hydrogen generated by corrosion, particularly in acid environments. Hydrogen embrittlement from this source may be accelerated greatly by the presence of certain catalysts such as sulfides and selenium. Bloom²⁰ has described examples of corrosion-generated hydrogen cracking in martensitic stainless steels and other hardened stainless alloys.

The second source of hydrogen is that generated by galvanic corrosion of a couple, with the hydrogen contaminating the cathodic member. Uhlig²¹ reported an example of this in which hardened 13 percent chromium steel was caused to fail rapidly by coupling to aluminum in 3 percent sodium chloride solution.

The third source of hydrogen is that generated by an impressed cathodic "protective" current. It is of interest that, whereas cathodic protection may be an effective means of preventing stress-corrosion cracking which involves an electrochemical mechanism, this same cathodic current may result in cracking of alloys which are susceptible to hydrogen embrittlement.

An allied problem of recent importance has been the hydrogen cracking of electroplated high-strength steels used in aircraft and missiles. An example, as-

sociated with cadmium plating, was described by Cash and Scheuerman.²²

Other Forms of Attack

The forms of attack listed under item D in Table 2 are special cases which do not fit readily under the other categories. Some of these forms may not be true cases of stress-corrosion cracking. But their recognition and description does have some importance.

The first of these cases is thought to be associated with the wedging action of oxides or other solid corrosion products. The idea is that these products are deposited in active cracks, that the resulting volume increase leads to a pressure build-up which develops lateral tensile forces, that these forces combined with residual and applied stresses cause crack extension, and that this permits entry of more corrosive constituents to continue the process. Nielsen²³ recently demonstrated experimentally that significant stresses do develop under such circumstances. For example, in one of his experiments, he found measurable stress build-up from hydroxides precipitated under constraint from an electrolyzed chloride solution of an austenitic stainless steel.

An area where wedging action has received considerable attention is that related to the effect of environment on the elevated-temperature creep-rupture properties of alloys. It has been recognized for many years that the environment, whether gaseous or otherwise, may have a detrimental effect on the rupture life and ductility of alloys subjected to stress at high temperatures. This subject was reviewed recently by Finnie and Heller.²⁴

It is of interest that the creep-rupture life of certain alloys is sometimes better in air than in vacuum or inert gases. This would seem contradictory to the oxide-wedge theory. One possible explanation is that the material is strengthened by local "alloying" in the form of internal oxidation and nitriding of the material in the vicinity of the crack. Examples of creep strengthening of this type have been described by Shahinian²⁵ and Widmer and Grant²⁶.

Item D-2 in the table covers failures in which the initial formation of a crack is associated with a corrosion process, but in which the crack then proceeds by purely mechanical means from the stress concentration produced by the initial defect. Whether or not the initial crack will propagate mechanically depends on the stress, geometry and material in accordance with the Griffith-Irwin theory²⁷. This theory states that rapid crack extension will occur whenever a small increase in crack area would result in the release of at least as much elastically stored energy from the piece as the energy required to form the crack. Experimental means to measure the "critical energy release rate" which controls crack propagation were described recently by Lubahn²⁸.

The final item in Table 2 is a type of failure which has been called stress-sorption cracking. Uhlig¹² cited examples of materials, including Mg₂Sn crystals

and certain Al-Zn alloys, which cracked in the absence of any known stress by exposure to pure water or salt solutions. The proposed mechanism was that certain ions, such as Cl^- or OH^- , adsorbed selectively at grain boundaries or other lattice imperfection sites, thereby leading to crack generation from the reduction of surface energy and loss of affinity between adjacent metal atoms.

Uhlig carried this theory further by suggesting that similar ion adsorption might occur at lattice imperfection sites induced by plastic deformation. If this action occurred conjointly with electrochemical action, it could result in a mechanism of crack propagation in which physical parting along paths of low surface energy would alternate with electrochemical dissolution of anodic areas. This type of crack propagation would fit in with the intermittent crack-growth pattern which has been observed and reported to be characteristic of several metals undergoing stress-corrosion cracking. Also, as metals may be selective in reception of adsorbed ions, the theory may account for the observation that stress-corrosion cracking occurs in certain electrolytes and not in others.

Closure

In conclusion, it would be fitting to quote Charles F. Kettering, who once said, "A problem thoroughly understood is usually fairly simple." This same quotation was used at the conclusion of the 1944 ASTM-AIME Symposium on Stress-Corrosion Cracking. Now, in 1960, it is interesting to see if general understanding of the nature of stress-corrosion

cracking is actually any more complete. Certainly the intervening sixteen years have helped to narrow the gap. But although progress has answered some of our older questions, it has also broadened our perspective and introduced new questions that still remain unanswered.

Thus, the problem is not yet a simple one. The author's only hope is that this paper has brought the reader some degree of order out of confusion, and a desire to carry on the search for truth and basic understanding.

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Any discussion of this article not published above will appear in December, 1961 issue.

Corrosion of Metals in Tropical Environments*

By A. L. ALEXANDER, C. R. SOUTHWELL and B. W. FORGESON

Introduction

THIS REPORT is the fifth in a series describing the corrosion rates of a variety of metals in the tropics. Earlier papers^{1, 2, 3, 4} have presented data for a number of unalloyed metals, structural steels, and wrought iron. As in earlier reports, the data presented here are based on the results of exposure of stainless steels lasting for eight years, during which measurements were made at the end of one, two, four, and eight years, respectively. Since the earlier work⁵ pointed to the inadequacy of the short-term data, it is proposed to continue these studies until data are obtained covering a total exposure period of sixteen years. The methods and procedures employed in the program are described in the first report of the series.¹

The Panama Canal Zone has within its 45-mile length a large variety of exposure conditions. Available here are deep jungles and extensive cleared areas; access to two oceans, the Atlantic with a surf beach and the Pacific with a normally clean shoreline; a large body of almost-constant-level fresh water in Gatun Lake; and a brackish water, constant-level lake at Miraflores. Practically all tropical exposures are found in this area, and test sites can be set up on military reservations or other secure locations serviced by good roads. Severe storms that might endanger years of testing are unknown. Thus, from all considerations, conditions are ideal for long-term natural-environment-exposure testing. The sites employed included marine and inland atmospheric exposure, fresh water immersion, and continuous and mean tide sea water immersion.

The discovery of stainless iron and steel about 1912 by Preasley, Strauss, and Manner and the introduction of these metals into the United States in 1914 were momentous events for the ferrous metals industry. The uses and production of these metals, which started as a trickle, developed so that by 1946, when

Abstract

Eight-year exposure tests of six stainless steels to underwater and atmospheric environments in Panama Canal Zone are described. Test environments included sea water immersion, lake water immersion, seashore inland, inland, sea water mean tide, and seashore. Structural steels, phosphor bronze and naval brass were also evaluated for comparison. Stainless steel plates later were galvanically coupled to carbon steel strips for testing in tropical waters.

Stainless steels were not recommended for sea water service where perforation of structure is a consideration. They gave excellent results, however, in tropical fresh water. Tropical atmospheres appeared more corrosive to stainless steels than did temperate climates.

4.2.7, 6.2.5

this corrosion program was started, the annual production of stainless steel ingots had reached 550,000 tons.⁶

Stainless steels have amazingly good corrosion resistance for certain conditions of exposure. These metals differ from most others in that they depend for corrosion resistance on surface passivity engendered by an oxidizing exposure medium, rather than upon natural chemical inertness. (Binder⁶ has stated that characteristics of both the stainless steel and the corrosive environment must be fully recognized in order to apply these steels successfully to a specific purpose.) When such facts are not ordinarily known, it is frequently necessary to evaluate the metals under operating conditions before an optimum alloy selection can be made. Since data concerning the long-term resistance of stainless steels to

natural environments were rare and since no knowledge existed concerning the effects of the more corrosive tropical environments, studies of stainless steels became an important component in the present investigation. Six different alloys of stainless steel were included in some or all of the five tropical exposures. Thus, results from the exposure of six stainless steels have been completed for eight years and are reported herein.

Experimental Conditions

Exposure Environments

Specific locations of each of the test sites have been described previously.¹ These sites provide for exposure to fresh water, sea water, mean tide, marine and inland atmospheres. The atmosphere and water at each were sampled^{2, 3, 4} sufficiently to gain some idea of any unusual corrosion agents which might be present. In addition, sufficient meteorological data were collected at each site to permit correlation with weather conditions described by the Panama Canal Company, Section of Hydrography and Meteorology,² to cover a minimum period of twenty years.

Metals and Methods

The six stainless steels studied in these natural tropical environments include: (A) a low-cost general-purpose martensitic type containing 13 percent chromium, AISI No. 410; (B) an easily formed

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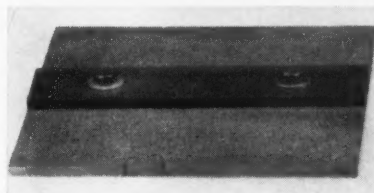


Figure 1—Underwater bimetallic couple sample.



Figure 2—Typical stainless steel (13-8) corrosion pattern after eight year's exposure in the Pacific Ocean, Panama Canal Zone.



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1954 of the Baltimore Section. He was chairman of the symposium on Organic Coatings for Tankers at the 1952 NACE National Conference, and a lecturer in NACE short courses at the universities of Tennessee and California.

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CHARLES R. SOUTHWELL is a Materials Engineer for the Naval Research Laboratory. Since joining the organization in 1954 he has been engaged in the study of deterioration of metals, woods, and protective coatings, in tropical environments. From 1950 to 1954 he was Engineer-in-Charge of concrete and protective coatings for the construction of the International Falcon Dam, and prior to 1950 was a Materials

Engineer for the Panama Canal Company doing investigative work pertaining to concrete, metals, and protective coatings for use in design of a sea-level canal at Panama. He attended St. Mary's University of Texas, and is a member of NACE.



B. W. FORGESON is in charge of the tropical exposure and laboratory facilities in the Panama Canal Zone for the Naval Research Laboratory. He has been in the employ of the laboratory since 1953, during which time he has been actively engaged in the study of corrosion, protective coatings and the biological deterioration of materials. From 1940 until 1952, he worked for the Special Engineering Division of the Panama

Canal as a materials engineer assigned to various research problems and laboratory work concerned with a proposed construction of the third set of locks and the sea level canal. He attended Pennsylvania State University and is a member of NACE.

ferritic alloy commonly used for atmospheric exposures containing 17 percent chromium, AISI No. 430; (C) a general-purpose austenitic stainless alloy of 17 percent chromium and 7 percent nickel, AISI No. 301; (D) the best known and most widely used of the stainless steels, the austenitic alloy containing 18 percent chromium and 8 percent nickel, AISI No. 302; (E) an 18 percent chromium,

13 percent nickel, and 2 percent molybdenum austenitic alloy, containing the high nickel and added molybdenum to give superior resistance to marine exposures, AISI No. 316; and (F) an 18 percent chromium and 10 percent nickel austenitic stainless steel, with $\frac{1}{2}$ percent titanium added to increase resistance to intergranular corrosion, AISI No. 321.

Of these stainless steels, A, B, C, and E were exposed in the tropical atmospheres at Miraflores and Cristobal. A phosphor bronze (G) and a typical low-alloy steel (J) were included at these sites for comparative purposes. Stainless steels A, D, E, and F were immersed in tropical waters at Fort Amador and Gatun, along with phosphor bronze (G), mild structural steel (I), and, in one instance, naval brass (H) as controls. Complete chemical composition and letter identification of all the metals included in this report are given in Table 1.

Ten replicates of each metal were exposed in each environment. This provided for the removal of duplicate panels from each location after one, two, four, eight, and sixteen years. Before exposure, all test pieces were solvent cleaned and vapor degreased in trichloroethylene. The test pieces were then dried in an oven at 160 F before being placed in a desiccator for cooling prior to initial weighing. Each sample was weighed, measured, inspected, and photographed before being installed in its rack.

Uncoupled samples for exposure at the immersion sites measured 9 inches square and $\frac{1}{4}$ inch thick. Bimetallic couples were $9 \times 9 \times \frac{1}{4}$ inch plates to which $2 \times 9 \times \frac{1}{4}$ inch strips were bolted. A view of an assembled couple is shown in Figure 1. All underwater test panels were in contact with ceramic insulators only and were oriented so that the rolling axis was horizontal. Each rack contained four sets of duplicate specimens, with glass barriers between each set and between the terminal specimens and the ends of the rack. This arrangement provided insulation against possible stray electrical currents. The racks were located so that significantly dissimilar metals would not be in proximate locations.

Upon removal from exposure, the immersion panels were cleaned at the site of marine growth and any heavy corrosion product. The panels from all locations were then chemically cleaned at the laboratory: stainless steels, bronze, and brass with 18 percent hydrochloric acid, and structural steels in 10 percent sulfuric acid with 1 percent quinoline ethiodide inhibitor. Cleaned samples were weighed, measured, inspected, and photographed. Pit depths were referred to the original surface of the metal, either by measurement from an uncorroded surface or by calculation using the original and final average measured thickness of the sample. The average of the twenty deepest pits represents the average of the five deepest pits measured on each side of duplicate specimens (an area of 2.25 sq ft for the immersion samples and 0.89 sq ft for the atmospheric samples). The methods of exposure and evaluation were considered in more detail in the first paper of this series.¹

Underwater Corrosion Results

Type of Corrosion Attack

Passive stainless steel is practically noncorrosive in natural environments. When corrosion does occur it results from a destruction of passivity in some area through contact with a nonoxidizing solution; for example, pits form at points of fouling attachment in sea water. These pits are often very deep and sometimes elongated; they are formed by the action of active-passive cells.

Figure 2 shows typical pitting attack occurring on austenitic stainless steels in tropical sea water. The side views show the heavy pitting on a vertical edge and a typical section through the plate. The elongation of the pits occurred in the direction of gravity and transverse to the direction of rolling, thus indicating that metal defects had no part in the growth of these pits. Uhlig⁷ explains the elongated pits in chloride solution as being caused by the formation of a high-density, acidic ferric chloride within the pit that breaks down the passivity of the metal directly below; therefore, pitting progresses in the direction of gravity.

The apparent randomness of pitting would lead one to expect an uncertain direction to the corrosion-time curves and poor reproducibility of results. This was the case when pitting depth alone was considered, as will be shown later; but measurement of weight loss, all of which was due to pitting corrosion, gave very good reproducibility, and for most cases, almost a straight line relationship with time developed after the first year. This would indicate that, although differing in frequency or depth, the total corrosion pitting in sea water varies very little from plate to plate on samples of this size.

Table 2 is a comprehensive tabulation of corrosion damage for all the metals reported here. Since weight loss for the stainless steels was due entirely to pitting attack, the greater portion of the stainless surfaces remained in their original condition. No real meaning is attached to such terms as reduction in thickness or average penetration, so they have been omitted. Likewise, the pitting factor has no significance for these metals and is not included.

Relative Intensity of Various Exposures in Sea and Fresh Waters

The fresh water of Gatun Lake, which proved to be a very corrosive environment for ordinary carbon steels,³ producing eight-year weight losses of 46 gm per sq dm, had absolutely no corrosive effect on the austenitic stainless steels and very little on the martensitic straight chromium type; the latter showed a weight loss of only 1.1 gm per sq dm after 8 years' exposure.

Mean tide exposure also proved mild for the stainless steels. Eight-year weight losses amounted to only 16 percent of that produced by continuous immersion in sea water. Pitting was much less intense at mean tide than during total immersion, even though a heavy barnacle-type fouling developed at this half-tide elevation. The deep and elongated pits that occurred on the austenitic steel con-

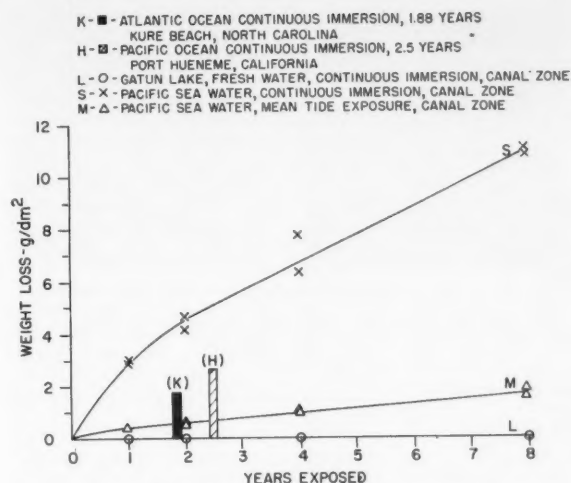


Figure 3—Relative corrosion of stainless steel (18-8) for immersed exposures in the Panama Canal Zone and on the East and West coasts of the United States.

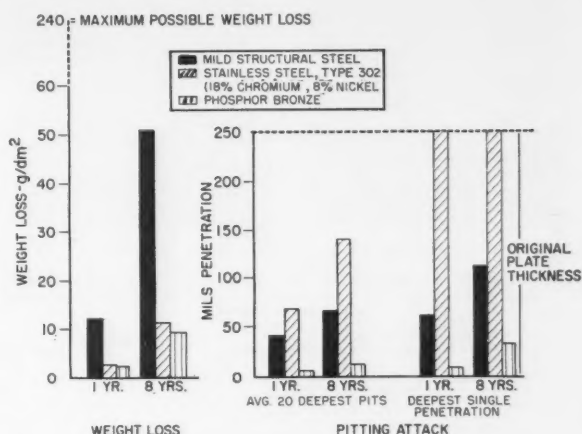


Figure 4—Comparison of stainless steel, phosphor bronze and structural steel, following continuous immersion in tropical sea water, with respect to weight loss and pitting corrosion attack.

TABLE 1—Chemical Composition of Metals Included in This Report

Metal	Type	Specification	Composition (percent)													
			C	Cr	Cu	Mn	Mo	Ni	P	Si	S	Ti	Fe	Pb	Sn	Zn
			Immersed Samples													
A	Stainless Steel No. 410 (13 Cr)	47-S-20 INT Grade 3A	0.07	13.15	0.08	0.48		0.23	0.016	0.29	0.021					
D	Stainless Steel No. 302 (18-8)	QQ-S-766 Grade 1C	0.09	17.79	0.072	1.15		8.88	0.034	0.56	0.018					
E	Stainless Steel No. 316 (18-13 & Mo)	QQ-S-766 Grade 1H	0.11	17.93	0.084	1.67	2.32	12.08	0.019	0.54	0.023					
F	Stainless Steel No. 321 (17-10 & Ti)	QQ-S-766 Grade 1I	0.08	17.63	0.13	1.11		10.33	0.013	0.44	0.030	0.50				
G	Phosphor Bronze	QQ-B-746 Grade A			95.54				0.25				0.010	0.048	4.21	0.30
H	Naval Brass	QQ-B-636 Type IV, Half hard			59.87								0.05	0.09	0.58	39.41
I	Mild Structural Steel	QQ-S-741 Type II, Grade A, Class 1	0.24	0.03	0.080	0.48		0.051	0.040	0.008	0.027					
			Atmospheric Samples													
A	Stainless Steel No. 410 (13 Cr)	QQ-S-766a Class 3a	0.08	14.42	0.078	0.44		0.52	0.017	0.35	0.020					
B	Stainless Steel No. 430 (17 Cr)	ASTM A 176-44 Grade 4 Finish 1	0.10	17.22	0.024	0.34		0.12	0.014	0.29	0.008					
C	Stainless Steel No. 301 (17-7)	QQ-S-766a Class 4 Condition a Finish 1	0.10	18.12	0.030	1.13		7.62	0.012	0.39	0.021					
E	Stainless Steel No. 316 (18-13 & Mo)	QQ-S-766a Class 5 Condition a	0.08	17.08	0.078	1.50	2.12	11.96	0.031	0.43	0.016					
F	Stainless Steel No. 321 (17-10 & Ti)	QQ-S-766a Class 6 Condition a	0.09	17.76	0.13	1.00		9.92	0.011	0.53	0.018	0.54				
G	Phosphor Bronze	QQ-B-746 Grade A			95.10	0.07			.358				.04	0.00	4.38	0.00
J	Low Alloy Steel	Proprietary Cu, Ni, Mn, Mo	0.087	0.03	0.59	0.73	0.19	0.82	0.065	0.067	0.018					

NOTE: Numbers shown for stainless steels are nearest equivalent AISI numbers with the approximate chromium or chromium-nickel content shown in parenthesis.

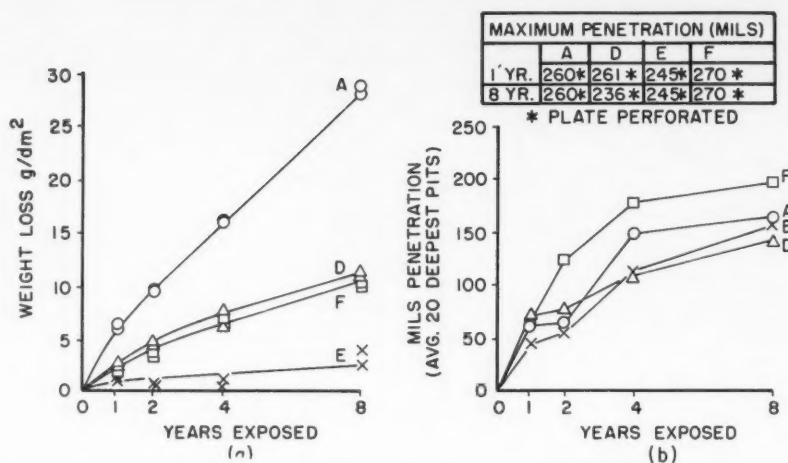


Figure 5—Comparative corrosion of various stainless steel alloys continuously immersed in tropical sea water. Code to stainless steel: A—Type 410 (13 percent Cr); D—Type 302 (18 percent Cr, 8 percent Ni); E—Type 316 (18 percent Cr, 13 percent Ni, and Mo); F—Type 321 (17 percent Cr, 10 Ni and 0.5 percent Ti).

tinuously submerged in sea water were not in evidence for the same metal at the mean tide location. Of the three austenitic stainless steels, all had perforated (250 mils) by the end of one year of continuous immersion in tropical sea water; for the same length of time, the

maximum penetration of similar plates at mean tide was only 26 mils. Even after eight years, penetration did not exceed 110 mils.

Continuous submersion in tropical sea water is a relatively severe exposure for stainless steels, as can be seen from the

above comparisons and from Figure 3. This figure shows the corrosion-time curves for 18-8 austenitic stainless steel in all three immersion environments. The bar graphs relate comparative losses of 18-8 alloy submerged continuously in temperate sea waters. Comparison with the corrosion curve from the tropical sea water in the Canal Zone shows that the samples from the East Coast of the United States, at Kure Beach, North Carolina⁸ suffered only 40 percent as much corrosion loss. The tests with the same alloy at Port Hueneme, California,⁹ indicated only half the corrosion damage as in the tropical sea water in the Canal Zone. Undoubtedly, the heavy marine fouling encountered in the tropics is a major cause of this greater corrosion damage.

Comparison of Weight Loss and Pitting of Stainless Steel, Structural Steel, and Phosphor Bronze in Tropical Waters

Gatun Lake fresh water is a relatively corrosive medium for structural carbon steel, causing weight losses approaching that occurring in sea water, but the 18-8 stainless steel and phosphor bronze are very resistant to both weight loss and pitting attack in this water. Thus, a con-

TABLE 2—Comprehensive Evaluation of Corrosion Damage of Stainless Steels, Structural Steels, and Phosphor Bronze Exposed to Tropical Environments in the Panama Canal Zone

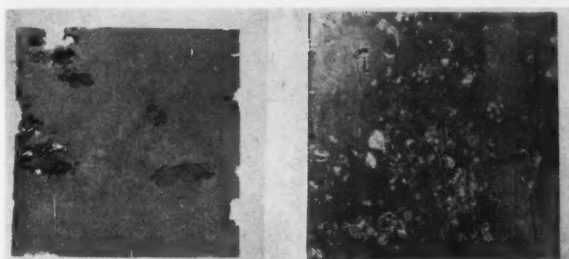
Metal	Type	Exposure	Weight Loss (g/dm ²)				Average 20 Deepest Pits (mils)*				Deepest Pit (mils)*				Percent Loss in Tensile Strength†	Type Corrosion Attack
			1 Yr	2 Yr	4 Yr	8 Yr	1 Yr	2 Yr	4 Yr	8 Yr	1 Yr	2 Yr	4 Yr	8 Yr		
A	Stainless Steel No. 410 (13 Cr)	Sea Water Immersion	5.99	9.56	15.87	28.09	61(11)	64(11)	148	161	260(p)	264(p)	260(p)	259(p)	**	KQH
		Sea Water Mean Tide	1.01	1.99	3.32	6.68	46	65	67	67	66	140	173	152	0	JKQ
B	Stainless Steel No. 430 (17 Cr)	Lake Water Immersion	0.00	0.18	0.65	1.06	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore Inland	0.08	0.11	0.11	0.08	0(0)	0(0)	3	0(0)	0(0)	0(0)	5	0(0)	0	KR
C	Stainless Steel No. 301 (17 Cr, 7 Ni)	Inland	0.05	0.07	0.07	0.07	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	KR
		Seashore	0.00	0.02	0.00	0.13	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
D	Stainless Steel No. 302 (18 Cr, 8 Ni)	Inland	0.00	0.00	0.01	0.01	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore	0.00	0.00	0.00	0.01	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
E	Stainless Steel No. 316 (18-13 & Mo)	Sea Water Immersion	2.93	4.43	7.06	10.99	70(12)	74	107	140	261(p)	238	286(p)	236	**	KQ
		Sea Water Mean Tide	0.26	0.55	0.99	1.76	7(13)	6(10)	26	57	16	20	82	110	0	JK
F	Stainless Steel No. 321 (17-10 & Ti)	Lake Water Immersion	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
G	Phosphor Bronze (Grade A)	Sea Water Immersion	1.18	0.65	0.54	4.08	44(7)	52	48	154	245(p)	82	93	245(p)	0	KQ
		Sea Water Mean Tide	0.13	0.10	0.27	0.39	5(9)	0(0)	7(12)	16	23	0(0)	22	30	1	JK
H	Mild Structural Steel (Pickled)	Lake Water Immersion	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
I	Low Alloy Structural Steel (Cu, Ni, Mn, Mo)	Sea Water Immersion	2.32	3.44	6.55	10.02	64(8)	120	175	193	270(p)	206	273(p)	272(p)	**	KQ
		Sea Water Mean Tide	0.26	0.40	0.67	1.34	8(11)	23	37	56	26	52	60	93	**	JKQ
J	Phosphor Bronze (Grade A)	Lake Water Immersion	0.00	0.00	0.00	0.01	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore	0.01	0.00	0.01	0.01	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	1	K
K	Mild Structural Steel (Pickled)	Inland	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
L	Low Alloy Structural Steel (Cu, Ni, Mn, Mo)	Sea Water Immersion	2.42	3.40	4.88	9.07	3(16)	10(16)	13	66	9	32	22	0(0)	2	ARH
		Sea Water Mean Tide	1.09	1.70	2.90	4.63	1(1)	0(0)	0(0)	0(0)	8	0(0)	0(0)	0(0)	3	E
M	Phosphor Bronze (Grade A)	Lake Water Immersion	0.27	0.56	0.70	0.10	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
		Seashore	0.43	0.63	0.91	1.37	0(0)	0(0)	0(0)	3(2)	0(0)	0(0)	0(0)	4	0	K
N	Mild Structural Steel (Pickled)	Inland	0.13	0.23	0.35	0.56	0(0)	0(0)	0(0)	3(1)	0(0)	0(0)	0(0)	3	0	K
		Seashore	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
O	Low Alloy Structural Steel (Cu, Ni, Mn, Mo)	Sea Water Immersion	11.81	16.55	31.39	50.88	41	47	66	66	62	61	113	86	12	AQR
		Sea Water Mean Tide	18.85	18.50	25.39	46.30	18(8)	16(7)	27(19)	40	27	23	55	65	10	RA
P	Phosphor Bronze (Grade A)	Lake Water Immersion	15.22	23.71	33.94	43.68	20	38	45	58	24	46	66	71	13	E
		Seashore	0.00	0.00	0.00	0.00	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0(0)	0	K
Q	Low Alloy Structural Steel (Cu, Ni, Mn, Mo)	Inland	3.43	4.71	5.94	9.74	8	7	5	12	14	12	6	24	2	AEH
		Seashore	2.09	3.51	5.08	6.86	—(0)	5(10)	5	12	—(0)	8	9	16	1	AEH

* Pit depths referred to the original surface of the metal either by measurement from an uncorroded surface or by calculation using the original and final average measured thickness of the sample. Average of 20 deepest pits represents average of the 5 deepest pits measured on each side of duplicate specimens. (Area, 2.25 sq ft on immersed specimens, and 0.89 sq ft on atmospheric specimens); values in parentheses indicate total number averaged when less than 20 measurable pits. Perforation of plate by deepest pit is indicated by (p).

† Percent changes in tensile strength calculated on basis of 1/4 inch thick metal (average of 4 tests for immersed specimens, average of 3 tests on atmospheric specimens).

Corrosion types are described in the Appendix of Part I. Those referred to here are: A—Uniform Attack, E—Localized Attack (Uniform), H—Concentration Cell, J—Marine Fouling Contact, K—No Visible Attack, Q—Pitting Attack (Random), R—Localized Attack (Random).

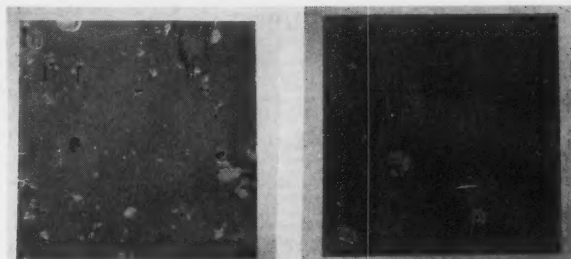
** Intensity and distribution of pitting prevented satisfactory tensile testing.



A Type 410 (straight chromium)

B Type 321 (high nickel)

Figure 6—Corrosion pattern for straight-chromium-alloyed and high nickel alloyed stainless steels, eight years under tropical sea water in Panama Canal Zone.



A Type 302 (18-8)

B Type 316 (molybdenum)

Figure 7—Improvement of corrosion resistance to tropical sea water with molybdenum-alloyed stainless steel, eight years under tropical sea water in Panama Canal Zone.

siderable advantage exists for the corrosion-resistant metals in this medium.

In sea water at mean tide, phosphor bronze exhibits the best corrosion resistance of the three metals considered, having no measurable pits and showing a weight loss of only 5 gm per sq dm in eight years. Stainless steel (18-8) was even better on the basis of weight loss alone, losing only 2 gm per sq dm. But pitting was considerable, and although no perforation occurred in eight years, the pits had progressed to 110 mils in depth. This was greater than the mild steel at mean tide, which was pitted to a maximum depth of 65 mils. The use of 18-8 stainless steel in the tidal range of the tropics should be limited to structures that would not be harmed by perforation, as such conditions are favorable for concentration and/or active-passive cell corrosion.

Figure 4 compares mild steel, 18-8 stainless steel, and phosphor bronze following continuous immersion in tropical sea water. The weight losses show the bronze and 18-8 stainless steel to be about equal in rate of corrosion while structural steel loses about five times as much weight in eight years. When depth of pitting is considered, however, the 18-8 stainless steel proves to be by far the most deeply penetrated, with phosphor bronze the least and mild steel intermediate between the two. Thus, for use in tropical sea water at low velocity, stainless steel containing 18 percent chromium and 8 percent nickel would not be satisfactory for any structure that would be damaged by deep and numerous pits. Other investigators^{5,10} have reported that in flowing sea water with velocities greater than five ft per sec little or no accelerated pitting of this stainless alloy takes place.

Comparison of Stainless Steel Alloys

Stainless steel containing 18 percent chromium and 8 percent nickel is the most widely used and best known of the stainless alloys. It reached this preeminence because of its corrosion resistance to many environments and its excellent mechanical and physical properties. Several other alloying elements have been added to the chromium-nickel austenitic steels to further increase their corrosion resistance under certain conditions. In sea water, 18-8 stainless steel is normally passive, but a borderline condition, such

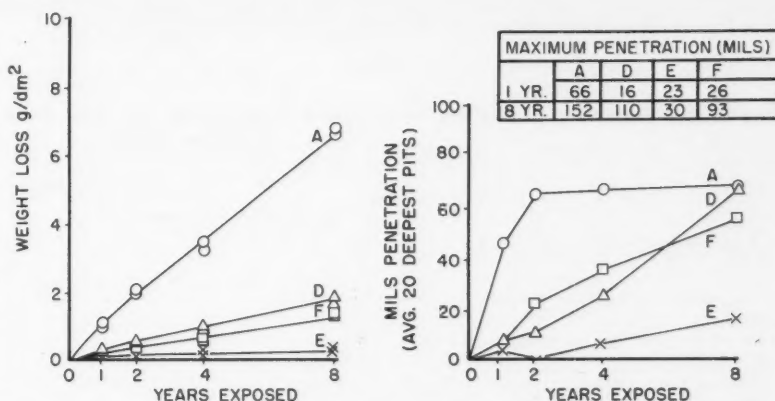


Figure 8—Comparative corrosion of various stainless steel alloys exposed to tropical sea water at half-tide elevation. Code to stainless steels: A—Type 410 (13 percent Cr); D—Type 302 (18 percent Cr, 8 percent Ni); E—Type 316 (18 percent Cr, 13 percent Ni, and Mo); F—Type 321 (17 percent Cr, 10 percent Ni, and 0.5 percent Ti).

○ A-1 STAINLESS STEEL TYPE 410 (A) 9" X 9" X 1/4" PLATE COUPLED TO 2" X 9" X 1/4" STRIP OF STRUCTURAL STEEL (1)
 × D-1 " " " " 302 (D) " " " " " " " " " " " " (1)
 △ E-1 " " " " 316 (E) " " " " " " " " " " " " (1)
 □ I-1 CONTROL, STRUCTURAL STEEL (1) SELF-COUPLED

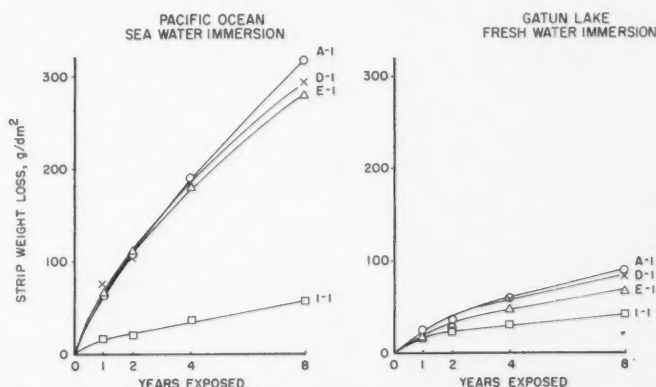


Figure 9—Comparative effect of three stainless steel alloys on the galvanic corrosion of ordinary structural steel in tropical waters.

as in the presence of heavy marine fouling attachment, could change it to the active state and trigger an accelerated corrosion attack. Molybdenum has been alloyed in these steels to increase the passivity range and thus enhance resistance to several media, including sea water. In some alloys small percentages of titanium or columbium are added to inhibit intergranular corrosion. Both mo-

lybdenum and titanium bearing austenitic stainless steel were included in the underwater exposures. The martensitic straight chromium steels are hardenable and less costly than the austenitic types. Martensitic 410 type with 13 percent chromium, was also studied in the submerged environments.

In Gatun Lake fresh water the three austenitic alloys, 302, 316, and 321, ap-

TABLE 3—Galvanic Corrosion in Tropical Waters of Couples Containing Relatively Large Areas of Stainless Steel

Couple	Corrosion Weight Loss (g/dm ²)*															
	Immersed in Pacific Ocean Sea Water								Immersed in Gatun Lake Fresh Water							
	Plate				Strip				Plate				Strip			
	1†	2	4	8	1	2	4	8	1	2	4	8	1	2	4	8
13 Cr (A) to (I) Mild Steel.....	0.3	0.0	0.0	0.2	65	109	192	318	0.0	0.0	0.0	0.0	24	37	59	90
13 Cr (A) to (G) Phosphor Bronze....	6.9	11	18	29	1.2	1.3	3.0	0.8	0.1	0.2	0.4	2.7	0.5	0.9	1.5	1.6
18-8 (D) to (I) Mild Steel.....	0.6	0.0	0.0	0.3	79	108	188	291	0.0	0.0	0.0	0.3	24	39	59	82
18-8 (D) to (G) Phosphor Bronze....	0.0	0.5	3.1	7.6	18	3.1	37	50	0.0	0.0	0.0	0.0	1.2	1.3	2.1	9.4
18-8 (D) to (D) 18-8.....	0.5	1.7	3.2	9.0	0.0	3.8	13	12	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
18-13 & Mo (E) to (I) Mild Steel.....	0.0	0.0	0.0	0.0	69	112	181	280	0.0	0.0	0.0	0.0	21	34	49	66
18-13 & Mo (E) to (G) Phosphor Bronze....	0.0	0.0	0.0	0.4	86	52	87	85	0.0	0.0	0.0	0.0	0.7	1.0	2.8	3.8

* Each value represents the average of duplicate samples.

† Years exposed.

TABLE 4—Galvanic Corrosion in Tropical Waters of Couples Containing Relatively Small Areas of Stainless Steel

Couple	Corrosion Weight Loss (g/dm ²)*															
	Continuously Immersed in Pacific Ocean Sea Water								Continuously Immersed in Gatun Lake Fresh Water							
	Strip				Plate				Strip				Plate			
	1†	2	4	8	1	2	4	8	1	2	4	8	1	2	4	8
18-8 (D) to (I) Mild Steel.....	0.3	0.0	0.0	0.0	15	21	35	54	0.0	0.0	0.0	0.0	16	25	37	50
18-13 & Mo (E) to (I) Mild Steel.....	0.0	0.0	0.0	0.0	16	20	35	56	0.0	0.0	0.0	0.0	16	25	38	50
18-13 & Mo (E) to (G) Phosphor Bronze....	0.0	0.0	0.0	0.0	4	5	8	13	0.0	0.0	0.0	0.0	0.4	0.6	0.9	1.6
18-13 & Mo (E) to (H) Naval Brass.....	0.0	0.0	0.0	0.0	8	10	15	20	0.0	0.0	0.0	0.0	0.9	1.6	2.2	4.1

* Each value represents the average of duplicate samples.

† Years exposed.

peared to be about equal and exhibited complete corrosion resistance during eight years' exposure. Martensitic Type 410 was almost as good, showing no measurable pits and a weight loss of only 1 gm per sq dm after eight years.

Results from continuous immersion in sea water are presented in the corrosion-time curves of Figure 5. The weight-loss curves in Figure 5a show that straight chromium, Type 410, lost 2.5 times as much weight as the standard 18-8. This accelerated loss was not due to an increased number of pits, but rather to the enlargement of a relatively small number of areas previously pitted, as shown in Figure 6. On the other hand, differences in weight loss in the three austenitic alloys are related directly to the frequency of pitting of the plates. In this respect there was no significant improvement with the slightly increased nickel content and titanium addition of Type 321. However, considerable improvement over 18-8 was shown by Type 316, which contains a higher nickel content (13 percent) and 2 percent molybdenum. Molybdenum stainless steel has been recommended by many authorities^{7,11,12} for its corrosion resistance in low-velocity sea water. In these tropical sea water tests, too, there was a definite improvement in corrosion resistance with this alloy. This may be seen in Figures 5a and 7a and 7b.

Upon consideration of the depth of

pitting shown in Figure 5b, the advantage for the molybdenum alloy disappears. In the case of each stainless steel studied in sea water, perforation of 1/4-inch plates had occurred by the end of the first year of immersion. Average pit depth, although slightly favoring the molybdenum alloy after one and two years' immersion, revealed no significant advantage for this metal after four and eight years' exposure. Surprisingly, the 321 alloy containing titanium showed the highest average pitting penetration of any of the four stainless steels for all periods of exposure beyond the first year.

Half-tide exposure has been shown earlier to be much less severe to 18-8 stainless steel than continuous immersion. The other alloys were also considerably more resistant to half-tide than to continuous immersion. On the average, only about 1/4 to 1/3 as much loss occurred at half-tide as for the immersed samples. Figure 8 shows the progression of corrosion damage with time for the various stainless alloys exposed to mean tide. Weight-loss order correlated well with the immersed specimens, Type 410 exhibiting the greatest loss and the molybdenum alloy, Type 316, showing the least.

None of the stainless steels were perforated at mean tide, even after eight years' exposure. In this environment the 18-8 alloy and the similar metal with 1/2 percent titanium performed approxi-

mately equally, showing roughly 1/3 the penetration that these metals experienced during complete immersion. The straight chromium alloy 410 pitted very rapidly the first year, but then penetration stopped, and additional corrosion was the result of new pit development and the extension of area of the established pits. Alloy 316, with its higher nickel content and 2 percent molybdenum, was definitely superior at mean tide. Pitting penetration was much less than for the other metals after eight years. The average of the twenty deepest pits was only 17 mils and the deepest single penetration 31 mils.

Galvanic Corrosion of Stainless Steels

Prediction of the galvanic action of the high-chromium alloys is more difficult than with most other metals because of their dual potential in the galvanic series. Their position depends on the passivity or activity of the metal surface, which is controlled by the nature of the contact medium. In the active condition stainless steel exhibits nobility approximating that of ordinary steel, but in the passive state it is one of the most noble of the structural metals. The problem is further complicated by the rapid polarization in some solutions of bimetallic cells containing stainless steel. LaQue¹⁰ points out that open circuit potentials are unreliable for prediction of the magnitude of galvanic corrosion of these metals

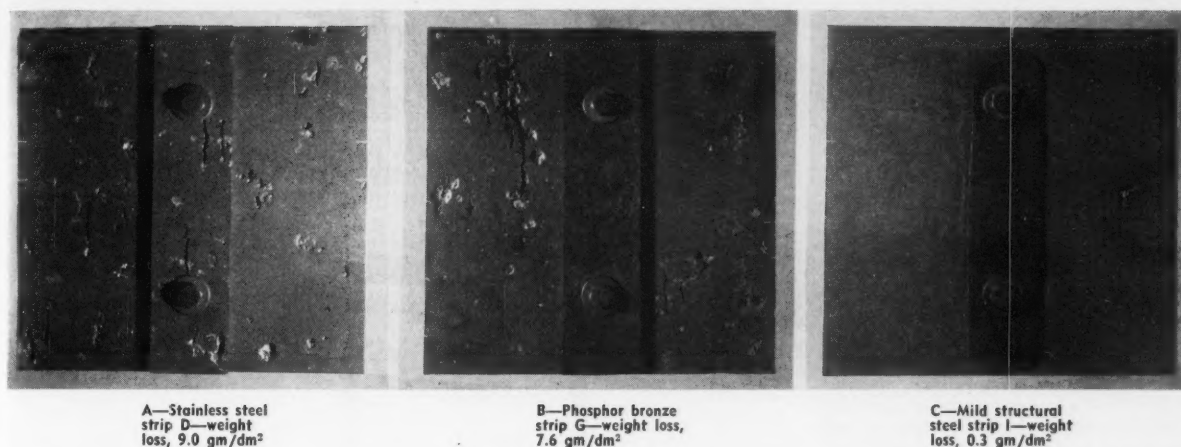


Figure 10—Protection of stainless steel (18-8) plates when coupled to relatively small strips of (A) stainless steel, (B) phosphor bronze, and (C) mild steel in tropical sea water for eight years.

under service conditions. He found that because of polarization in flowing sea water the effective nobility of 18-8 stainless steel was reduced to such an extent that it caused only $\frac{1}{4}$ the galvanic corrosion of carbon steel anodes as was caused by the normally less noble copper.

In the sea water near the Canal Zone and in the fresh water of Gatun Lake there were no water current factors to be considered, as the maximum rate of flow was less than $\frac{1}{2}$ ft per sec. The coupled and immersed samples, as shown in Figure 1, had a strip-to-plate area ratio of 1:6.5. Complete results for all couple combinations studied employing stainless steel as either the strip or plate are given in Tables 3 and 4.

In sea water the galvanic effect on the carbon steel strip from the stainless plate was considerable, as can be seen in Figure 9. Stainless steel 18-8 caused five times as much weight loss in eight years as a comparable carbon steel strip coupled to a plate of the same metal. In fresh water the effect was naturally much less, but was still apparent with the 18-8 producing twice the weight loss. The galvanic corrosion of carbon steel when coupled with the three different stainless alloys was not appreciably different in magnitude but was interesting because of the order of effect. After eight years the straight chromium, Type 410, seemed to have the most noble potential. The Type 316 alloy, with molybdenum, was seemingly the least cathodic, and standard 18-8 fell somewhere in between. The fresh water results indicated the same nobility order as the sea water tests. This order was exactly opposite from that predicted by open circuit potential measurements.

A naval brass plate-carbon steel strip combination showed even higher strip weight losses, averaging 350 gm per sq dm for eight years in sea water, even though brass is definitely a less noble metal than any of the passive stainless steels. These results indicate that a greater degree of polarization has occurred in the couples of stainless steel and carbon steel than for the brass-carbon steel combination. The reverse

order of the stainless steels also probably can be attributed to different degrees of polarization.

Even under the unfavorable conditions of low-velocity sea water and profuse development of marine fouling, complete protection from corrosion in tropical sea water was afforded all three of the stainless alloys by coupling to the carbon steel strip at the 1:6.5 anode-cathode area ratio, Figure 10. Recommendation for safe protection of stainless steel with carbon steel has been given as 1:1 in the literature.¹³ Phosphor bronze strips at the 1:6.5 ratio also were protective to stainless steel plates, but the amount of protection varied from none to almost complete, depending on the stainless alloy used. Type 410 had about the same potential as bronze and corroded at its normal rate. As shown in Table 3, the D alloy was slightly protected while the Type 316 was almost completely protected in this bimetallic combination. During the first year of exposure the bronze portion of the 316 couple had lost 35 times its normal uncoupled weight loss.

Atmospheric Corrosion Results

Relative Intensity of Inland and Sea Coast Locations

The Caribbean shore test site on the Washington Hotel roof, with its offshore breakwater and prevailing sea wind, is about 1.6 times as corrosive to carbon steel as the inland site located at Miraflores.² Ambler and Bain¹⁴ in their excellent study of corrosion in the tropics point out the extreme importance of airborne salinity and proximity to ocean surf in atmospheric corrosion. In the Canal Zone studies of stainless steels, the atmospheric exposures included three of the alloys tested underwater, (Types 410, 316, and 321). Also exposed to the atmosphere was Type 301, which is very similar to the underwater Type 302 and Type 430, 17 percent chromium.

Both the tropical inland and marine atmospheres were practically noncorrosive to all the stainless steels; the highest losses were recorded for Type 410 at the

marine site. They amounted to only 0.08 gm per sq dm for the first year of exposure and did not exceed 0.11 gm per sq dm during eight years' exposure. Binder¹¹ reports results on this same alloy for 540 days in a marine atmosphere at Wilmington, North Carolina and 600 days in an industrial atmosphere at Niagara Falls, New York. His results show 0.11 gm per sq dm loss for the marine site and 0.25 gm per sq dm for the industrial atmosphere. The three austenitic stainless steels in the Canal Zone atmospheres suffered no significant corrosion damage for eight years, at either inland or seacoast sites. There were no measurable pits on any surface, and the maximum weight loss obtained was only 0.01 gm per sq dm.

The tropical atmosphere thus appears to be no more corrosive to stainless steels than temperate climates, and the difference between seacoast and inland effects in the Canal Zone is minor, with the marine site a slightly more corrosive environment.

Comparison of Stainless Steels with Low-Alloy Structural Steel and Phosphor Bronze

The excellent atmospheric corrosion resistance of all the stainless steels can be seen in the bar graph presentation of four-year and eight-year results in Figure 11. As with the underwater results, comparisons have been made with phosphor bronze and a structural steel. In this case low-alloy structural steel was substituted for plain carbon steel, the low-alloy metal being a much more logical choice for atmospheric exposure. In the tropical atmosphere the high-chromium and chromium-nickel stainless steels were, as expected, much better in all respects than the low-alloy steel. Comparison with phosphor bronze was also favorable to stainless. The tensile strength losses of stainless 316 and 321 are only one percent and not considered significant at this time. Pitting of the two metal types was zero, except for one panel of Type 410 for which a depth of five mils was recorded. Weight losses of all stainless steels were zero or practically zero, while

the loss in weight of phosphor bronze, although small, increased steadily and revealed that some measurable corrosion was proceeding on this metal.

Any of the stainless alloys included here should give very satisfactory atmospheric corrosion resistance in the tropics, but for the use of stainless steels in any tropical environment precautions should be taken to eliminate the existence of cracks, holes, or crevices, where the collection of passivity-destroying agents can bring about accelerated pitting attack.

Conclusions

1. Eight-year immersion studies of various stainless steel alloys in the tropical waters of the Panama Canal Zone disclose that the stainless steels are severely pitted during sea water immersion, and the mean tide location produced only one-fourth to one-tenth the total damage; the metals are practically unattacked during immersion in fresh water.

2. Comparison of sea water attack on 18-8 stainless steel reveals that twice as much corrosion occurred in tropical sea water as was found at Port Hueneme, California, and two and a half times as much as at Kure Beach, North Carolina. The greater pitting attack in tropical sea water is probably caused by the very heavy marine fouling attachment which interrupts the passivity of stainless steels and initiates active-passive cell corrosion.

3. Deep elongated gravity-guided pitting attack occurred on all the high-chromium-nickel austenitic alloys; the martensitic stainless without nickel corroded in an entirely different manner. This leads to the supposition that nickel may have a role in the formation of heavy acidic chlorides that cause the elongated attack.

4. Because of its vulnerability to pitting attack in sea water, 18-8 stainless steel is inferior to phosphor bronze for this service; pitting depth was also greater than that found for ordinary carbon steel. Stainless is not recommended for sea water service where perforation of structure is a consideration.

5. Stainless steels give excellent results in tropical fresh water, where they were found to be much superior to ordinary steels and equal to phosphor bronze, which is practically noncorrosive in this environment.

6. Of the four stainless steels exposed underwater, the alloy containing 18 percent chromium, 13 percent nickel, and 2 percent molybdenum was superior in the sea water studies, but even this superior stainless steel was perforated in just one year in tropical sea water. The frequency of pitting was reduced by the alloying constituents, but when pits did

A - STAINLESS STEEL NO. 410 (13% CR)
B - " " " 430 (17% CR)
C - " " " 301 (17% CR, 7% NI)
E - " " " 316 (18% CR, 13% NI & MO)

F - STAINLESS STEEL NO. 321 (17% CR, 10% NI & TI)
G - PHOSPHOR BRONZE
J - TYPICAL LOW ALLOY STRUCTURAL STEEL

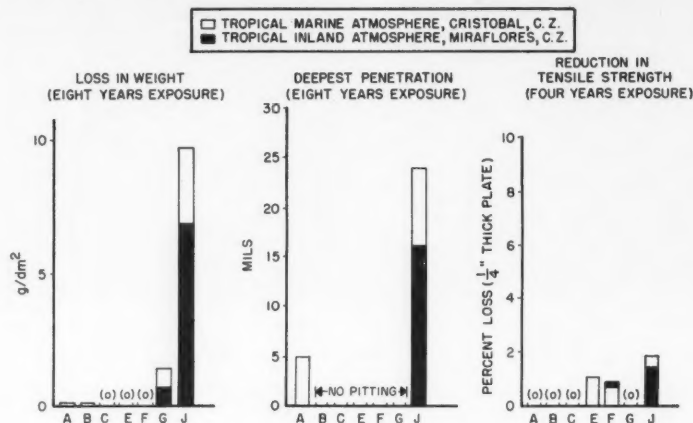


Figure 11—Comparison of corrosion damage of stainless steel, phosphor bronze, and low-alloy structural steel in tropical environments.

become established the rate of penetration was unchanged.

7. Galvanically, stainless steel plates coupled to carbon steel strips at a 6.5:1 area ratio caused a considerable increase over the normal corrosion of the carbon steel strips in tropical waters. In sea water the increase amounted to five times as much weight loss and in fresh water approximately twice as much. The effective difference of the three different stainless steels on the plain carbon steel was slight.

8. Complete protection of all the stainless steel plates was afforded by the coupling of carbon steel strips at a 6.5:1 plate-to-strip ratio. A phosphor bronze strip for this same ratio accelerated the corrosion of the stainless alloyed with 13 percent chromium, protected slightly the 18 percent chromium and 8 percent nickel stainless, and gave complete protection to the 18 percent chromium, 13 percent nickel, and 2 percent molybdenum stainless steel.

9. For the stated conditions the tropical atmospheres were practically non-corrosive to stainless steels, while bronze suffered some weight loss at both the inland and seacoast exposure stations; structural low-alloy steel was both pitted and reduced in thickness in both these atmospheric environments.

Acknowledgments

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Any discussion of this article not published above will appear in December, 1961 issue.

Effect of Mineral Impurities in Water On the Corrosion of Aluminum and Steel*

By LEONARD C. ROWE and MONTE S. WALKER

Introduction

WATER IS often used without regard to mineral impurities and their possible effect on corrosion. A geological survey of public water supplies in the United States showed that 81 percent of the people are supplied by surface water which is more variable in composition than ground water although containing less mineral matter.¹ Many impurities can be removed at water treatment plants, but 39 percent of the cities use no treatment or only chlorination. It is recognized that the chloride ion promotes corrosion, and the concentration of this ion in finished (tap) waters covered by the survey emphasizes the overall difference in waters.

Figure 1 shows that the chloride content ranges from zero to above 500 parts per million. Although most waters have less than 50 parts per million of chloride, 10 percent of the population uses water at a higher concentration. It is important to understand the effect of mineral impurities on corrosion.

The electrical conductance method was selected to study this problem because of its simplicity and the rapidity with which results can be obtained. This method has been discussed in detail by other authors and appears to be well suited to this purpose.^{2,3,4} Although pits which perforate the metal prevent an absolute corrosion rate measurement, it has been observed that loss of metal by pitting is reflected in the measurement, and the method is useful for comparative studies of this nature. Visual observations are reported in the appropriate section to augment conclusions drawn from corrosion measurements.

It was necessary to determine the limitations of the method, and its effectiveness in reflecting variations in corrosion. Steel was selected for this purpose because a small amount of corrosion is readily reflected by a change in metal conductivity. Both steel and aluminum were used in studying the effect of mineral impurities on corrosion, but the major emphasis has been placed on aluminum.

Experimental Procedures

Specimen Preparation

Typical specimens were prepared by cutting strips from SAE 1020 carbon steel or 1145H aluminum shim stock with a metal cutter. The final dimensions of a steel specimen were 0.001 x 0.125 x 14 inches. The aluminum specimen was 0.003 inch thick, but it retained the other dimensions. The specimen was cut down

Abstract

A laboratory study was made to determine the effect in water of single or mixed impurities on the corrosion of aluminum or steel. This study utilized the conductance technique for determining variations in corrosion rate. This method is based upon the concept that a change in the electrical conductivity of a thin metal strip reflects the corrosion which has occurred.

The effect of several common mineral constituents in water was determined. These include such impurities as chloride, sulfate, bicarbonate, carbonate, calcium, magnesium, and silicate. Of this group, it was found that chloride and sulfate are most detrimental to steel. No single impurity affects the corrosion of aluminum to the degree that mixed impurities do. A solution containing soluble salts of copper, chloride, and bicarbonate has an accelerating effect on the corrosion rate of aluminum. Pitting depends upon impurity concentration and temperature.

4.6.6, 6.4.2, 6.2.3

the middle at both ends to provide four electrical leads, two inches long. It was necessary to coat with wax a one-half inch portion of each steel lead at the juncture of the lead and main strip to prevent excessive corrosion at this point.

Each specimen was degreased in acetone and mounted in an epoxy holder, having four holes to separate leads. The leads were firmly attached to threaded posts with washers and nuts. The specimen strip was formed in the shape of a loop which fitted the test container without touching the sides.

Steel specimens were chemically cleaned for 30 seconds in a 10 percent sulfuric acid solution at 66 C. This was followed by rinsing in distilled water, a brief immersion in a 0.5 percent sodium phosphate solution at 82 C, repeated rinsing in distilled water, and a final rinse in ethyl alcohol. It was essential that the holder as well as the specimen be immersed in distilled water to remove residues of the cleaner.

Aluminum specimens were chemically cleaned for 30 seconds at 66 C in a solution containing 25 grams per liter sodium phosphate, 15 grams per liter sodium silicate, and 7 grams per liter sodium carbonate. The chemical cleaning was followed by repeating rinsing in distilled water and finally in ethyl alcohol.

The specimen holder was made to fit firmly in a 200-ml pyrex test tube. Before cleaning specimens, the test tube was filled with 180 ml of solution and transferred to a constant temperature water bath, capable of maintaining a constant temperature to ± 0.01 C. Nine tests could be run at one time.

The cleaned specimens were transferred to the test container in the bath with a minimum loss of time. An aerator, made from 3-mm glass tubing, was inserted through a hole in each holder.



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Electrical leads to the potentiometer were attached firmly to the four posts on each holder. The solutions were aerated at a rate of 10 cc per minute which permitted a modest agitation of the solution. The initial potential measurement was taken after an elapsed time of one-half hour. All determinations were run in triplicate and the results averaged.

Conductance Measurement

The electrical conductance of a specimen was measured at the beginning of the experiment and at periodic intervals thereafter. The measurement consisted of passing a known amount of current through the metal and measuring the voltage drop across the specimen.

The measuring circuit was similar to that described by Dravnieks and Cataldi.² A direct current of 0.3-0.5 ampere from a 6-volt acid storage battery was passed through the specimens and a manganin reference resistor of 0.5 ohm which was immersed in silicone oil and contained

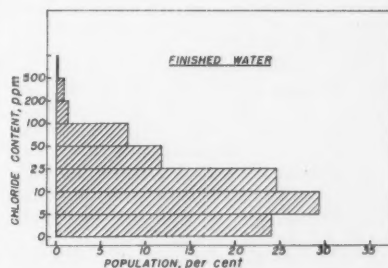


Figure 1—The difference in the chloride content of waters used by a representative population of the United States.

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TABLE 1—Comparison Between the Actual Weight Loss of Steel and That Calculated from Conductance Measurements

SOLUTION	WEIGHT LOSS, MILLIGRAMS		
	Actual	Calculated	Difference
Distilled Water 1.....	9.2	8.6
2.....	9.2	9.0
3.....	9.8	9.4
Average.....	9.4	9.0	-0.4
*Sodium Chloride 1.....	6.8	7.3
(one percent) 2.....	6.4	6.3
3.....	7.7	7.8
Average.....	7.0	7.1	+0.1
*Sodium Chloride 1.....	13.6	14.2
(one percent) 2.....	13.2	14.0
3.....	13.1	13.6
Average.....	13.3	13.9	+0.6

* Triplicate tests were terminated at different time intervals.

in the same bath. The potential was measured with a potentiometer having a sensitivity of 0.01 mv. A selector switch was used which permitted the measurement of potential across any specimen or the reference resistor.

The current was adjusted with a rheostat to a value which resulted in a convenient potential drop across the specimen. The potentiometer was switched to the reference resistor and the potential measured. This potential was directly proportional to the current through the specimen and hence directly proportional to the conductance. Potential measurements were taken at periodic time intervals, using the same initial potential drop across each specimen as the standard. The change in potential across the reference resistor represented the change in conductance across the specimen. The conductance was directly proportional to the cross-sectional area of the specimen since the length was constant. Any relative change in width was small compared to that in thickness because of the use of thin metal strips. Hence, a decrease in area may be assumed to be due to a decrease in thickness.

For purposes of calculation, the appropriate potential drop across the reference resistor can be substituted for that of conductance. Since comparative differences in corrosion are of interest in this study rather than absolute corrosion rates, the percent corrosion was used and was calculated as follows:

$$\text{Percent Corrosion} = \frac{(C_0 - C)}{C_0} \times 100$$

where C_0 equals the initial conductance and C the measured conductance at any time.

Experimental Results

Method Reliability

The reproducibility of a given potential with time, using the described technique, was determined by immersing steel specimens in n-heptane or silicone oil at 26 C. The potential reading was reproducible after 13 days to within 0.01 mv in the n-heptane and 0.03 mv in the silicone oil. No greater deviation was observed during this period.

An additional check on the validity of results obtained by conductance measurements was made by comparing calculated weight loss with actual weight

loss. The results are shown in Table 1. Two different test solutions were used, one being highly conductive and the other relatively non-conductive. These results substantiate the reliability of the method although many pin hole pits were developed in the salt solution. The maximum variation between the calculated and actual weight loss, as shown in the table, is 0.6 mg and the minimum 0.1 mg, both based on results from the conductive solution but at different time intervals. The maximum variation for any single reading is 0.8 mg.

The effect of solution conductivity on specimen conductance measurements was determined in another manner. The normal procedure was followed, and strips were immersed in a 1 percent sodium chloride solution. After the measurement of each potential in this solution, the strips were transferred to a non-conductive benzol solution, held at the same temperature, and the potential measured. The conductance measurements obtained from the two solutions were used to calculate weight loss. The difference between the two methods of measurement was never greater than 0.2 mg which is within the experimental error of the method.

Steel

It was necessary to be somewhat arbitrary as to the amount and type of impurity to be investigated. Major constituents in water were chosen and used in concentrations which might be expected to be detrimental. The effects of many of these impurities are summarized in Table 2. Each value in the table represents the average of three determinations. The effects of chloride and sulfate ions are similar. Mixtures of the two ions produce about the same amount of corrosion as either ion alone at equivalent concentrations. Chloride ions have an immediate effect on corrosion at a concentration as low as 3 ppm with a continued effect up to 100 ppm and a leveling off above this point. Although the effect of the sulfate ion was not determined at a concentration below 50 ppm, it is assumed that it would react in a similar manner. Corrosion rate curves are uniform and are essentially straight lines through 48 hours. It is difficult to extend the time beyond 48 hours because of the rapid corrosion rate in the salt solutions.

Silicates inhibit corrosion as would be

TABLE 2—Effect of Various Mineral Impurities on the Corrosion of Steel at 26 C

Mineral Impurity*	Percent Corrosion In 24 Hours At Various Concentrations Of Impurity			
	0	50 ppm	100 ppm	300 ppm
None.....	2.6
Sulfate.....	12.2	14.1	17.8
Chloride.....	11.1	14.1	19.6
Sulfate-chloride (1:1).....	13.4	14.2	18.9
Silicate.....	0.02	0.01	0.02
Bicarbonate.....	7.5	12.4	14.4
Carbonate.....	5.4	11.3	0.03
Calcium-Magnesium (1:3).....	14.3	13.4	14.3

* All impurities were added as sodium salts with the exception of calcium and magnesium which were added as sulfates.

expected. Bicarbonates and carbonates are very similar in that they produce an increase in corrosion until a concentration of 300 ppm is reached. At this point the corrosion rate in the carbonate solution drops to practically zero. This is probably due to pH since there is a more rapid rise in pH in the carbonate solutions. Calcium and magnesium ions, added as sulfates, have no greater effect on corrosion than an equivalent amount of sulfate ion added as the sodium salt. Thus, the hardness ions do not appear to contribute to any increase in corrosion rate.

Based on these results, it can be concluded that chloride, sulfate, bicarbonate, and carbonate are the ions which promote most effectively the corrosion of steel at 26 C. Different results may be obtained with mixed ions in solution or with a longer period of exposure.

Although there was no specific intent to study inhibitors, the effect of chloride or sulfate ions on borax inhibition was included in this investigation. Table 3 lists the results that were obtained. They show that the borax concentration needed to provide protection is dependent on the amount of chloride or sulfate ion in solution. In a general sense the contribution by either ion is about the same although the chloride ion appears to be somewhat more critical at higher concentrations and the sulfate ion at lower concentrations. The National Chemical Laboratory of England reported that the benzoate inhibitor is equally susceptible to chloride and sulfate ions.⁵ Past experience has indicated that borax and benzoate are somewhat similar in this respect, and these observations are tentatively supported by the results from the two laboratories. It was also reported that similar corrosion rates were obtained in chloride and sulfate solutions without inhibition. This agrees with the results shown in Table 2.

Aluminum

The corrosion rate of aluminum was low in aerated distilled water at 26 C. There was no substantial increase in corrosion rate when either chloride, sulfate, bicarbonate, or calcium was added at concentrations as high as 300 ppm, or copper up to 2 ppm. The addition of a combination of any two of these ions did not produce a substantial increase in corrosion rate. The combination of chloride, bicarbonate, and copper ions in the presence of air produced a significant increase

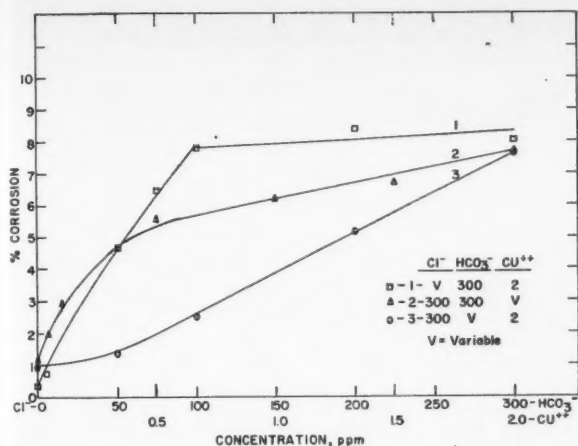


Figure 2—Corrosion of aluminum at 26 C after 72 hours exposure in water solutions containing chloride, bicarbonate and copper ions.

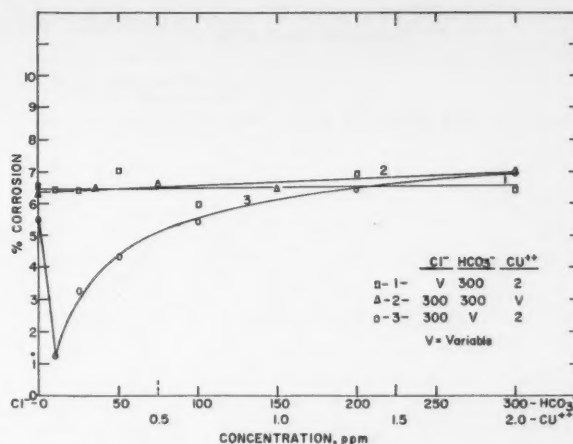


Figure 3—Corrosion of aluminum at 71 C after 72 hours exposure in water solutions containing chloride, bicarbonate and copper ions.

in the corrosion rate of aluminum. In the absence of air, only one-third the amount of corrosion occurred. Although copper is not a normal impurity in municipal waters, it may be present in solution because of the corrosion of copper-bearing alloys in a water system.

The combined addition of chloride, bicarbonate, and copper salts to distilled water was selected for a more detailed investigation. It was found that a near-maximum contribution was obtained from each ion at concentrations of 300 ppm chloride, 300 ppm bicarbonate, and 2 ppm copper. Chloride and bicarbonate were added as the sodium salts and copper as the chloride. The results, obtained by varying the concentration of a single component from this combination and maintaining the other two at the near-maximum level, are shown in Figure 2. The chloride ion produces a rapid initial rise in corrosion up to a concentration of 100 ppm, and little additional effect is obtained with higher concentrations. The copper ion has its greatest effect on corrosion up to a concentration of 0.5 ppm and continues to show a gradual effect up to 2 ppm. Copper has been added in concentrations as high as 10 ppm with no additional effect on corrosion. This may be due to a tendency of the copper to precipitate at these higher concentrations. The bicarbonate ion produces a gradual increase in corrosion with an increase in concentration.

A pitting type of corrosion was normally observed at 26 C. The following observations were made relative to the formation of pits. Gas bubbles collected on the aluminum strip soon after immersion, and they appeared to be an indication of pit sites. Mounds of corrosion products developed with time and grew in size. Gas bubbles continued to rise from the center of the mounds. The mound showed a gradual decrease in growth with time. After removal of the corrosion product, a pit was found with a bright ring around it. The pits were large in diameter but few in number at low concentrations of either chloride, bi-

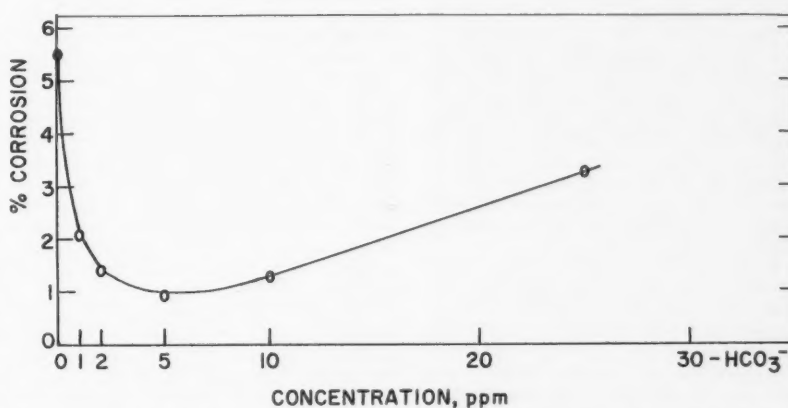


Figure 4—Effect of the bicarbonate ion at low concentrations on the corrosion of aluminum at 71 C. A concentration of 300 ppm chloride and 2 ppm copper ion was used in all solutions.

carbonate, or copper ion. With an increase in concentration, the pit density increased and the pits became smaller. In many cases the pits had penetrated through the metal.

The effect of chloride, bicarbonate, or copper ion as single additions was explored at 71 C. Copper or chloride solutions produced no increase in corrosion over distilled water. Bicarbonate solutions produced a gradual increase in corrosion with an increase in concentration. This is associated with oxide-film formation. No pitting occurred in any case. The combination of two ions in solution produced a significant effect only for the specific combination of copper and chloride. At a constant copper concentration of 2 ppm, the chloride concentration was varied. The corrosion rate was relatively constant until the chloride concentration exceeded 100 ppm, and at this point the rate began to rise. Pitting was noted only at chloride concentrations above 200 ppm. The substitution of the sulfate ion for the chloride ion produced no similar effect on corrosion.

The effect of the combination of chloride, bicarbonate, and copper ions on aluminum corrosion was repeated at 71 C.

The results of these tests are shown in Figure 3. The corrosion is relatively constant at all chloride or copper ion concentrations. The bicarbonate ion produces the greatest effect on corrosion. The percent corrosion drops as bicarbonate is increased from zero, reaching a minimum at approximately 10 ppm, and gradually rises again with concentration. The portion of the curve showing the minimum was investigated further and the results are shown in Figure 4. The decrease in corrosion is gradual, reaching an actual minimum at 5 ppm of bicarbonate.

Although the measured amount of corrosion appears to be comparable at the temperatures of 26 C and 71 C, the type of corrosion was different. No pitting occurred at 71 C unless bicarbonate was absent. Pitting occurred only in solutions containing chloride and copper ions. The addition of 5 ppm of bicarbonate stopped the pitting and inhibited corrosion. The metal remained bright. Further additions of bicarbonate produced a uniform white deposit on the metal surface. This deposit has been identified by X-ray diffraction as β -aluminum oxide tri-hydrate. Thus, the bicarbonate ion appears to assist in the

TABLE 3—Effect of Chloride or Sulfate Ions on the Borax Inhibition of Steel at 26 C

Ion Concentration, ppm	Percent Corrosion In 24 Hours At Various Concentrations Of Borax			
	0.1%	0.25%	0.5%	1.0%
Chloride 50.....	7.5	0.9	0	0
100.....	2.1	0.2	0
300.....	6.9	1.2	0.1
500.....	2.7	0.2
1000.....	1.2
Sulfate 50.....	13.1	0	0	0
100.....	0.2	0	0
300.....	10.8	0	0
500.....	0.7
1000.....	5.7

formation and growth of the oxide film. The data obtained at various time intervals showed a logarithmic film growth which varied with time and bicarbonate concentration. At 48 hours the growth rate was logarithmic above 100 ppm, at 72 hours above 50 ppm, and at 96 hours above 25 ppm.

The effect of the bicarbonate ion at two different temperatures can be summarized in this respect. Pitting occurs at 26 C in the presence of chloride, bicarbonate, and copper ions. The size of the pit decreases and the pit density increases with an increase in bicarbonate concentration. At 71 C, pitting occurs only at zero bicarbonate, inhibition occurs at 5 ppm, and uniform film formation occurs above 25 ppm.

To determine the effect of temperature on these reactions, three solutions were used at temperatures of 41, 56, and 64 C. All solutions contained 300 ppm chloride and 2 ppm copper. The bicarbonate concentration was zero in one solution, 5 and 300 ppm in the other two respectively. These are the concentrations at which the differences in corrosion were greatest. The results of these tests are shown in Table 4. There is a steady increase in corrosion with temperature at zero bicarbonate. Large pits are formed at 56 C and above. At 5 ppm bicarbonate, the corrosion reaches a maximum at 41 C and decreases above this temperature. Pitting occurs only at 56 C. The degree of corrosion is about the same at 300 ppm bicarbonate for all temperatures, but many very small pits are formed at 56 C and below but none above this temperature. It is concluded that the change in effect occurred in a temperature range in the vicinity of 50 C. The change was gradual, and competing reactions probably occurred in this range.

The addition of calcium as the chloride to solutions containing bicarbonate, chloride, and copper ions produced no effect at any temperature until 300 ppm calcium was added. At this point a precipitate formed which reduced the bicarbonate to the range where inhibition occurred. The loss of bicarbonate was verified by acid titration. A similar effect could have been obtained by adding 5 ppm bicarbonate initially as in the other experiments.

Discussion

Porter and Hadden, Davies and others have discussed a proposed mechanism

for the pitting corrosion of aluminum.^{6,7,8} Copper or other cations which have a low hydrogen overvoltage are considered necessary for pit initiation but not for growth. It has been qualitatively determined in this laboratory that copper did deposit on the metal surface, thus satisfying the requirements for pit initiation. During pit initiation, hydrogen evolution is predominant from copper sites. Eventually, the cathodic reaction becomes the reduction of oxygen, indicating the need for dissolved oxygen. The peptizing action of the chloride ion is presumed by Aziz to prevent effective film formation.⁹ The chloride ion, because of its size and mobility, can diffuse through weak points in the oxide film. Thus, the conditions for pit initiation and initial growth are satisfied by the presence of oxygen and chloride and copper ions.

The auto-catalytic nature of a pit is dependent upon maintaining an acid solution in the pit. The acid prevents the formation of an insoluble aluminum hydroxide and maintains the metal in an active condition. Hagyard and Santhiapillai have proposed that the auto-catalytic process is not due to low pH of the pit solution but to the formation of aluminum chloride which can activate passive aluminum.¹⁰ Regardless of the exact nature of the solution in the pit, it tends to be acidic. There is a normal tendency for the alkali formed at the cathode to neutralize this acidity. It has been postulated that the bicarbonate neutralizes the cathodic alkali, preventing it from destroying the acid formed at the anode. It has been stated that calcium is necessary for the occurrence of nodular pitting, which is a specific type associated with the formation of isolated mounds of insoluble aluminum hydroxide where pitting occurs under the mound. This study has not shown this need although a direct comparison cannot be made because of the variation in specimen thickness and exposure time. A type of pitting similar to nodular pitting has been observed in the presence of copper, chloride, and bicarbonate ions, and oxygen at specific temperatures. The concentration of bicarbonate ion and the temperature of the solution appear to have a major influence on these reactions.

When pits are formed, hydrogen gas is evolved and mounds of corrosion products are formed at a few isolated sites. Upon removal of the corrosion products, bright-metal rings are observed around

TABLE 4—Effect of Temperature on the Corrosion of Aluminum at Various Bicarbonate Concentrations.⁽¹⁾

Bicarbonate Concentrations, Parts Per Million	Percent Corrosion In 72 Hours At Various Temperatures				
	26 C	41 C	56 C	64 C	71 C
0.....	0.98 *	2.70 *	3.92 —a few large pits—	4.43	5.50
5.....	1.02 *	4.33 *	3.28 a few small pits	1.00 *	0.93 *
300.....	7.65 —many very small pits—	7.14	7.00	7.43 *	6.94 *

(1) 300 ppm chloride and 2 ppm copper ion in all solutions.
* No pitting.

the pits. The pits are large and few in number. This type of pitting appears to be due to the suggested reaction of alkali neutralization at cathodic sites. The number of pit sites increases and the size of the pit decreases at high concentrations of bicarbonate, indicating that acidity at anodes is neutralized by the bicarbonate. The reaction is stifled by oxide-film formation.

The growth and character of the oxide film is the determining factor in the reaction. The film growth rate increases with temperature and is stimulated by the bicarbonate ion. This ion is presumed to be essential for film formation since in its absence the pitting increases with temperature. This is due to the increased activity of the chloride ions which diffuse through weak spots in the film. The decrease in dissolved oxygen at the higher temperatures may also be a factor in poor film repair. Very small concentrations of bicarbonate are sufficient to enhance film growth and deter the action of chloride ion. There is evidence of pit initiation under these conditions, and it may be that a small concentration of bicarbonate interrupts the acid formation at local anodes, permitting oxide-film growth. Any further addition of bicarbonate at these temperatures merely stimulates the formation of an oxide film.

The results show that two competing reactions occur. In one case, the oxide film repair is poor and the chloride ion can easily penetrate the film to stimulate corrosion at pit sites. In the other case, the bicarbonate ion enhances film repair which prevents further activity. Both effects are temperature dependent, with the former being predominant at temperatures below approximately 50 C and the latter above this temperature.

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Oxidation of Iron-Chromium Alloys at 750-1025C*

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Introduction

AN UNDERSTANDING of the relatively good high temperature resistance to oxidation displayed by the stainless steels can be gained by learning in detail about the oxidation behavior of the simpler systems: pure iron, iron-nickel alloys, iron-chromium alloys. In recent years many such studies have been conducted with the results that (1) the oxidation of pure iron is about as well understood as any other chemical reaction, (2) the gross features of iron-nickel alloy oxidation are reasonably well characterized, (3) the oxidation of iron-chromium alloys is still relatively poorly understood.

Point No. 3 is considerably more complex than the other cases. The purposes of this work are to present additional experimental evidence of several types on the oxidation of a series of iron alloys containing chromium in amounts between 0.2 and 10 wt. percent, to attempt to account for some of the behavior observed, to define more precisely problems for future investigation, and to suggest possible functions for chromium and nickel in the high temperature oxidation of stainless steels.

Early studies of the oxidation of iron-chromium alloys showed the general decrease in rate with increasing chromium content,¹ the general morphology of the products,¹ intermittent accelerating rates (break away),¹ and concentration of the chromium near the alloy-scale interface,² possibly accompanied by depletion of chromium in the alloy surface.³ Criteria for alloy protection were suggested: (1) a continuous, adherent layer near the metal with a low solubility for iron oxides and a low diffusivity for iron ions,² (2) the oxide must have good mechanical properties and the alloying element must diffuse rapidly enough in the alloy phase to maintain the protective scale composition,⁴ (3) inclusions or local formation of scale containing a high proportion of the oxides of iron might contribute to scale rupture.⁵

Even careful attention to surface preparation could not eliminate the accelerated oxidation steps on 18-8 stainless steel.⁶ The time for breakaway on AISI stainless steels 304, 410 and 430 increased as the oxygen pressure or temperature

Abstract

The rates of oxidation of iron alloys containing 0.2 to 10 percent chromium were measured at 750 to 1025 C. The nature of the corrosion products was investigated by metallography, x-ray diffraction and chemical analysis. At very low chromium concentrations there was a small increase in the oxidation rate compared with pure iron for short times, but for longer times the rate diminished. The oxidation rate at a given temperature diminished with increasing chromium concentration. The rates were not given by any simple rate law, presumably because the products contained many pores and cracks. The accelerations in rate during an isothermal measurement did not occur at reproducible times or average thicknesses, which suggested that scale fracture played an important role. A mechanism for scale embrittlement by chromium was proposed. The ways in which chromium might contribute to a reduced rate of oxidation of iron were discussed.

6.2.2, 3.5.9, 3.7.2

decreased and the chromium or nickel content of the steel increased. It was proposed that spinel formed first with rhombohedral oxide growing mainly after breakaway. Consequently spinel was considered to be more protective.⁷

For long term oxidation runs on commercial steels containing approximately 12, 16 and 27 percent Cr and very little nickel, Caplan and Cohen⁸ reported no wüstite derivatives for the temperature range 870 to 1100 C. Yearian, Randell and Longo⁹ found no wüstite at or below 825 C for 6 percent Cr, none below 1000 C for 14 percent Cr and none as high as 1160 C for 17 and 26 percent Cr alloys. The chromite was always in the innermost layer in contact with metal. Spinel farther out contained less chromium and was close to Fe_3O_4 in composition. The total spinel layer was found to contain about the same ratio of Cr to Fe as the alloy. The small percentage of manganese in these steels seemed to play an important role in the reaction. MnCr_2O_4 was a constituent of one type of scale. Silicon and perhaps molybdenum may also have had an effect out of proportion to their small concentrations.

In order to rationalize this complex process, it seems necessary to start from the constituent equilibria and transport processes, with two guiding principles. Only oxygen is added and nothing is removed from the systems, so all metal atoms no longer in the alloy must be in the oxide. Each change must bring the system, both generally and locally, closer to equilibrium. Two questions, not clearly answered by earlier work, stand out: (1) Does cation separation by differential reaction at the interfaces or diffusion in the scale layers lead to depletion of the alloying element in the metallic phase, (2) Is the breakaway phenomenon reproducible, suggesting some sort of process under diffusion control, or erratic, suggesting a more structure-sensitive

process like crack initiation or nucleation of a new phase?

Experimental

Rate of Oxidation Measurements

Rectangular plates approximately $\frac{3}{4} \times \frac{1}{4} \times \frac{1}{16}$ inch were cut from iron (Puron), and iron-chromium alloys containing 0.20, 0.34, 2.00, 4.35, 4.71 and 9.73 weight percent chromium were vacuum melted from electrolytic iron and electrolytic chromium. The 4.71 and 9.73 percent Cr alloys are often designated hereafter as 5 and 10 percent for convenience. The rates of oxidation of higher chromium alloys, 15 to 25 percent Cr, proved to be too slow to measure satisfactorily in the authors' apparatus, and the results of experiments with alloys of these compositions are not reported here.

Polished and degreased specimens were held in a spring balance furnace,¹⁰ hydrogen treated and oxidized for selected times at selected temperatures. Puron plates of the same size and shape were run for comparison. Because of edge and corner effects resulting from the shape, the pure iron rates do not agree exactly with earlier results. They are used here only as control runs and do not supplant the earlier results.

The rates for representative runs are shown in Figures 1-7. The departures from parabolic behavior appeared to be more pronounced the higher the chromium content and the lower the temperature. The oxidation rate, especially the initial rate, seemed to be very sensitive to pretreatment of the surface. The results, even for identical preparation, were not very reproducible. For that reason no attempt has been made to show all of the many rate curves which were measured. Less than one quarter of the runs are shown in Figures 1-7. Some qualitative observations based on all measurements are given below.

In Figures 1 to 4 rate curves for pure iron and 0.20, 2.00, 4.35 and 8.97 percent Cr alloys are compared at 850, 900, 950 and 1000 C. The irregularity of many of the alloy curves and the crossing of many curves for different compositions are common. For the curves in Figures 5 to 7 the experimental points were taken so close together that they are omitted from the plots. Figure 5 is a later series (than Figure 1) of measurements at 850 C for Fe and 0.20, 0.34, 4.71 and 9.73 percent Cr alloys. Rate curves for 0.20 percent Cr alloys at temperatures ranging from 800 to 1025 C are shown in Figure 6.

Most of the runs in this study were carried out directly on specimens ground through 600 silicon carbide paper and de-

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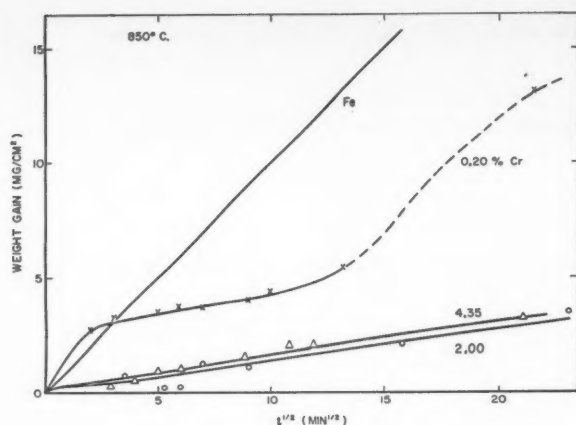


Figure 1—Oxidation rate curves for Fe and 0.20, 2.00 and 4.35 wt. percent Cr alloys in oxygen at 850 C.

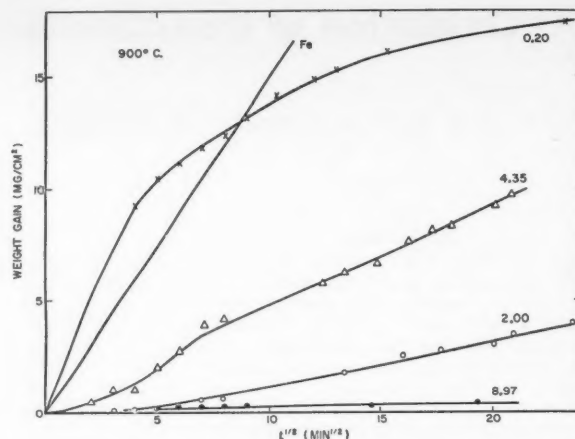


Figure 2—Oxidation rate curves for Fe and 0.20, 2.00, 4.35 and 8.97 wt. percent Cr alloys in oxygen at 900 C.

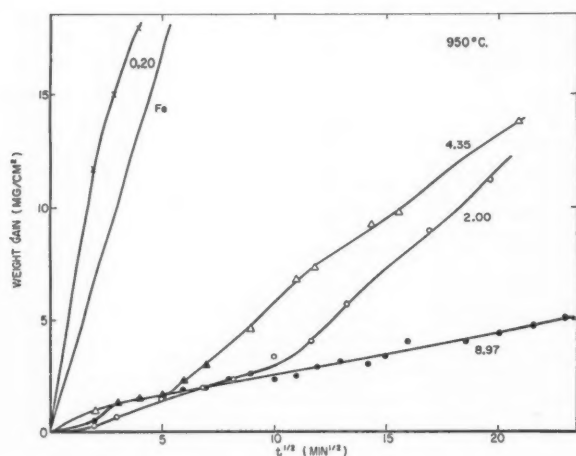


Figure 3—Oxidation rate curves for Fe and 0.20, 2.00, 4.35 and 8.97 wt. percent Cr alloys in oxygen at 950 C.

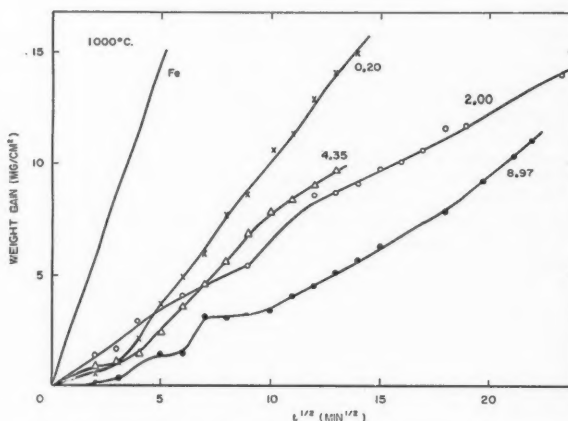


Figure 4—Oxidation rate curves for Fe and 0.20, 2.00, 4.35 and 8.97 wt. percent Cr alloys in oxygen at 1000 C.

greased. A treatment in hydrogen, purified by passage through a Deoxo tube, preceded the oxidation run. In the case of pure iron such a treatment cleans the surface very effectively and leads to highly reproducible results. In the case of these dilute chromium alloys it is evident that irregular curves are still obtained. From a comparison of the three runs at 950 C in Figure 7, where the time of heating in hydrogen at the oxidation temperature prior to oxidation is given on each curve, it appears that the longer the hydrogen treatment the faster the initial rate of oxidation and the slower the long term rate.

The products of oxidation showed a great deal of porosity as well as some cracks and wrinkles. It is not justifiable to give the rate measurements for all such samples in detail. A few general observations are summarized here.

Cr additions of 0.2 and 0.34 percent result in reduction of the long term constants (weight gain divided by the square root of the time) by a factor of about 3 ± 1 with respect to pure iron. The reproducibility from sample to sample was much poorer than the overall precision in

measuring time, weight change and temperature (in the range 670 to 1025 C). Wiggles in the curve were more frequent and more pronounced at lower temperatures. Often the initial rate was faster than that for pure iron (see Figures 1-3).

At 950 C, six 0.2 percent Cr samples scattered over rates differing by a factor close to three. Initially four, perhaps five, samples oxidized faster than pure iron, but by 100 minutes all had become slower than pure iron. With increasing time it appeared that the rates would become increasingly slower compared with iron.

Five percent Cr alloy samples at 700 and 750 C oxidized about half as fast as 0.34 percent samples initially, but the oxidation rate dropped off very rapidly with time. At 800 C one 5 percent Cr specimen started to oxidize more rapidly than pure iron but decreased very rapidly in rate.

At 850 C, 10 percent Cr alloys do not differ from 5 percent alloys more than the latter differ from each other. All lie

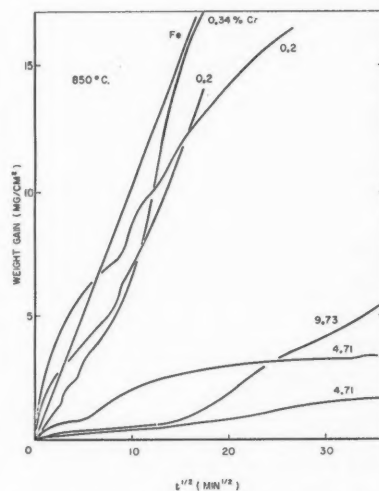


Figure 5—Oxidation rate curves for Fe and 0.20, 0.34, 4.71 and 9.73 wt. percent Cr alloys in oxygen at 850 C. The densely taken points are omitted.

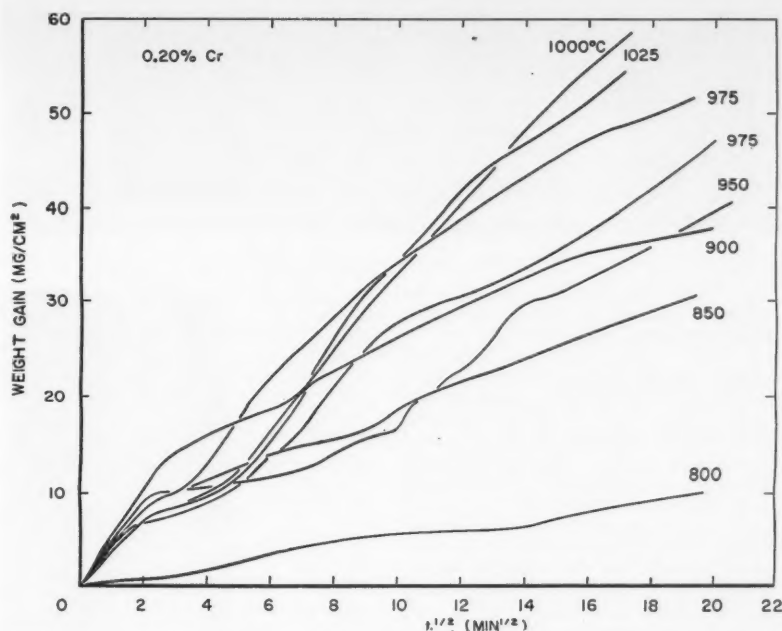


Figure 6—Oxidation rate curves for a 0.20 wt. percent Cr alloy in oxygen at temperatures between 800 and 1025°C.

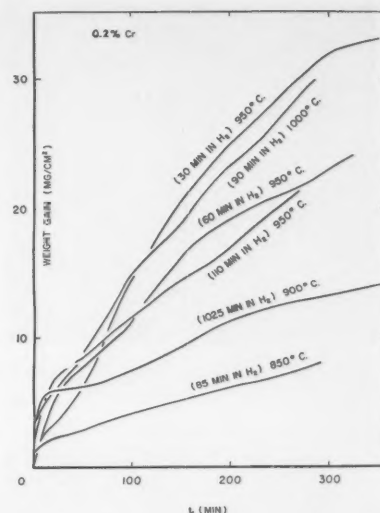


Figure 7—Oxidation rate curves for a 0.20 wt. percent Cr alloy pretreated in hydrogen at the oxidation temperature for the time stated on the curve.

below 0.2 and 0.34 percent samples by a factor of about 6.

Except possibly for a brief initial period which shows a maximum length and rate for samples of about 0.2 percent Cr, the effect of chromium is to reduce the oxidation rate of iron. Chromium also seems to promote formation of pores and cracks within and between the oxide layers, perhaps by increasing the brittleness of the products. The pores and cracks which are not connected to the reactant gas volume should contribute to the reduction in oxidation rate and may be largely responsible for the deviations from the parabolic rate law. In any case, the breakaway phenomenon could not be made reproducible.

Metallographic Observations

Wüstite is designated here as W. The spinel oxides are designated collectively by S, where the composition lies between Fe_3O_4 and FeCr_2O_4 . Rhombohedral solid solutions with compositions between Fe_3O_4 and Cr_2O_3 are designated R. In all samples examined the highly reflective R phase shows changes in reflected intensity when rotated between crossed polarizers. All photomicrographs are printed in such a way that the metal phase, whether visible or not, lies on the left, and the metal-oxide interface is vertical.

Pure iron—Figure 8, for a specimen reduced in hydrogen then oxidized for one hour at 846 C, shows heavily etched metal, a crack, W and S layers of about equal thickness, and strange R protuberances covering only part of the surface. The proportions of the phases are not typical, but the picture is shown because of the unusual hematite morphology.

0.2 percent Cr—Figure 9 shows a moderately porous layer of spinel grown in 253 hours at 840-843 C on a surface pre-

treated for 3.5 hours in hydrogen. There is no evidence of W. The product is mainly S with a little R on the external surface.

Figures 10 and 11 are scales grown for 18 hours at 927-930 C on a surface pretreated for 3 hours in hydrogen. In Figure 10 the R layer is about one fourth as thick as S. There is apparently a small amount of R at the inner irregular edge of S. The S layer is moderately porous. Figure 11 has a very small amount of R on the outer surface and some R growing into S along intruding cracks. The mottled region occupying the inner half of the S layer looks like the outer half under bright field illumination. Only under polarized light does the mottling become distinctly visible, hence it is not undistorted, cubic spinel entirely. The nature of the aggregate is not completely clear, but only R and S are indicated by x-ray diffraction. It may be that R of moderately high Cr content is precipitated on cooling. Examination at very high magnification suggests a poorly defined intergrowth type of morphology. Along the alloy surface a thin, irregular oxide exists, separated by a wide crack from the balance of the scale. The oxide is probably S predominantly with a composition somewhat different than that of the thicker layer.

The sample shown in Figure 12 was oxidized 4.7 hours at 1000 C. The W layer has S precipitate in the outer two thirds of its thickness. It is separated by a large crack on the outer side from the irregular S layer and on the inside by another crack under which a thin, bright, continuous S layer overlies another W layer which adheres to the metal.

0.34 percent Cr—Pretreated 20 hours in hydrogen and oxidized for 24 hours at 781 C, the specimen in Figure 13 shows



Figure 8—Pure iron reduced in hydrogen, then oxidized one hour at 846 C. Fifteen percent nital etch, polarized light, approximately 140X.

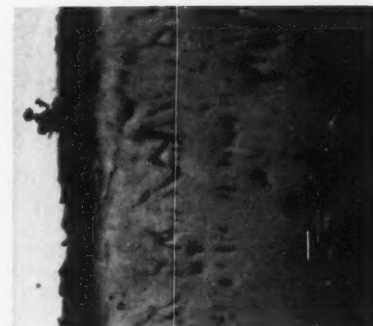


Figure 9—0.2 percent Cr alloy held in hydrogen for 3.5 hrs. then oxidized 253 hrs. at 842 C. Unetched, approximately 160X.

only a thin S layer with traces of R on the outer surface and around pores. Figures 14-17 are taken from a specimen pretreated for 22 hours in hydrogen and oxidized 72 hours at 818 C. Figure 14 is unetched and Figure 15 is of the same



Figure 10—0.2 percent Cr alloy held in hydrogen for 3 hours then oxidized 18 hours at 930 C. Unetched, approximately 140X.

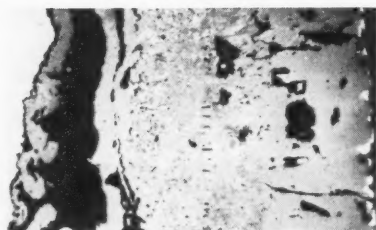


Figure 11—Same specimen as Figure 10. Polarized light, approximately 110X.

area etched to show the W layer more effectively with its S precipitate. The homogeneous appearing S layer is considerably thicker than the W, and the R outer layer is about one fourth as thick as S. Between W and metal there is a layer of rather uniform thickness, also seen in Figure 14, which is not identified. It does not polish or etch like any of the normal constituents, but in Figure 16 at higher magnification it looks porous or powdery and possibly consists of two phases, so it may not involve new phases. The layer shows no changes in reflected intensity when rotated between crossed polarizers. Figure 16 also shows subscale in the alloy and some detail of the pores in the inner part of the W layer. Figure 17 shows a very porous area in the S layer, which occupies a relatively great fraction of the thickness at this point. The outer R layer is reasonably sound. Small islands of S, perhaps of different composition than the main S layer, are growing between W and metal.

The 850 C specimen, Figure 18, pretreated 16 hours in hydrogen, oxidized 103 hours, shows mainly W containing S in greater quantity toward the outside and high porosity, both large and small, toward the metal. The thin S layer is very porous, and the R layer is very thin. Figures 19 and 20 are for a sample treated for 17 hours in hydrogen and oxidized 27 hours at 900 C. Figure 19 looks much the same as Figure 18, except that there is less fine porosity at the inner edge of the W layer, and Figure 19 is etched to show the distribution of S in W more clearly. An inner layer, like that in Figure 16, seems even more like a mixture of phases in Figure 20. Sizable blocks of S grow into the W layer just above the inner

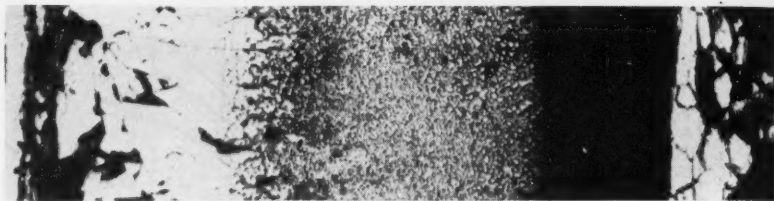


Figure 12—0.20 percent Cr alloy oxidized 4.7 hours at 1000 C. Etched in 10 percent nital, 200X.

layer. Note that the proportion of the thickness occupied by W increases with increasing temperature.

Two percent Cr—The sample in Figure 21 was oxidized for 3.4 hours at 950 C. An inner W layer with S precipitated in it is closely adherent to the alloy over most of the interface. Outside that layer the very porous W has veins of S parallel to the interface. Then there is a thick S layer, and finally an R layer. The alternating veins are presumably the result of cracks parallel to the interface which connected with the oxygen atmosphere at least for a time. A possibility not yet tested is that S was the product initially in contact with the alloy with W forming later as a result of chromium depletion.

Five percent Cr—The sample for Figures 22 and 23 was oxidized at 747 C for three days before a runaway furnace carried it to about 1250 C for nearly 14 hours. Microscopy and x-ray diffraction indicate only S and R as products. The inner porous mixture of nearly uniform thickness (Figure 22) may be derived from the inner layer seen in Figures 16 and 20. The mottled effect in the inner quarter of the S layer is seen in both Figures 22 and 23. At high magnification, Figure 24, the morphology suggests that R is forming with S during growth or, more likely, from S on cooling. An outer R layer exists, and R is seen around cracks and pores in the S layer. This is one of the samples on which electron microprobe analyses were obtained (see below).

X-ray Diffraction

On most specimens only surface reflection patterns were taken. Practically all of these showed only S, R and, when the layer was thin, alloy lines. More detailed mention is made here only of the few cases in which other procedures were followed.

The scale was mechanically pried off one 5 percent Cr alloy oxidized 12 hours at 850 C. Diffraction from the surface thus exposed gave strong R lines, weak S lines and very strong alloy lines.

Another 5 percent Cr specimen (treatment described for Figures 22 and 23) had its outer oxide layer mechanically separated. Both pieces were mounted and diffraction patterns were taken from successive faces as the layers were ground away, each in five steps. Starting from the outer surface the S and R lines, the only certain lines visible, indicated R decreasing progressively back to the position of the crack where S predominated strongly. Inside the crack R became strong again,

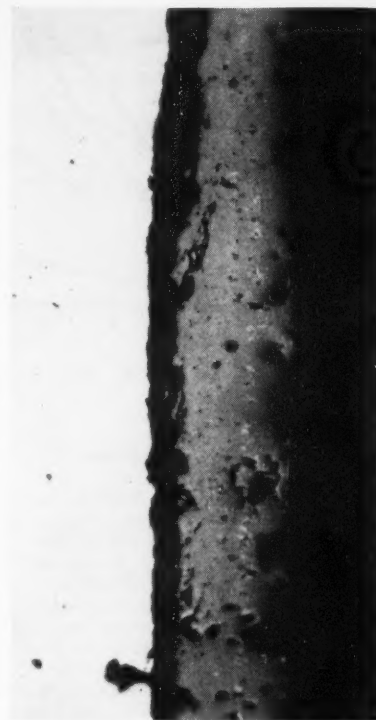


Figure 13—0.34 percent Cr alloy held in hydrogen for 20 hours, then oxidized 24 hours at 781 C. Unetched, 200X.

S weak, until alloy lines became most prominent. The observation supports the notion that R precipitates in the S layer on cooling, and that the inner layer contains no new phases.

W was observed only on a 0.34 percent Cr specimen from which the outer scale had partially chipped away. Apparently the chromium radiation did not penetrate far enough to reveal deep-lying W in the other cases.

Electron Microprobe X-ray Analysis

Two 5 percent Cr alloys were examined with the Cameca x-ray microanalyzer at the International Nickel Co. Research Laboratories by Drs. Kenneth Carroll and Sekyu Ohh. Because of the absence of suitable standards the compositions are only approximate, but quite adequate to show the relative effects sought. For the alloy phase only, the starting composition itself provided an absolute standard. Results for the same specimen as Figures 22 and 23 are shown in Figure 25. Figure 26 is a photomicrograph of the 5 percent Cr alloy oxidized at 850 C showing carbon deposition spots

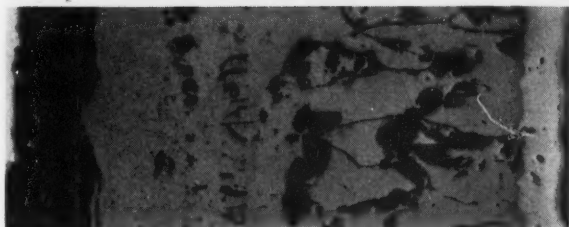


Figure 14—0.34 percent Cr alloy held in hydrogen for 22 hours, then oxidized 72 hours at 818 C. Unetched, approximately 75X.

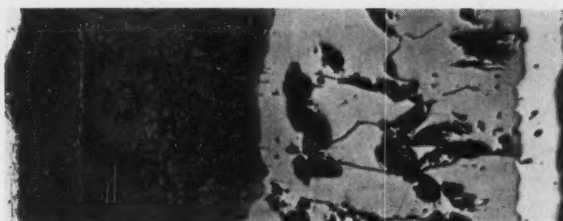


Figure 15—Same specimen as Figure 14 etched to show S precipitation in the W layer. Etched in 4 percent nital, approximately 75X.

where analyses were made. Figure 27 was taken from the opposite face. The specimen could not be polished for these pictures without removing the carbon spots, so some dirt accumulated in handling is evident on the surfaces. Figures 28 gives the results of the analyses.

The phase identifications in Figure 25 are based on x-ray and microscopic examination and in Figure 28 on microscopic examination only, although x-ray reflections from the surface of the latter specimen gave strong hematite lines, weak magnetite lines and very strong iron lines, consistent with the microstructure. In the mottled region the minor constituent, presumably R, seems to have the higher Cr content, because the high intensities appeared in spurts as the specimen stage was moved under the electron beam.

Discussion

The products of the oxidation of iron-chromium alloys are all structurally related to the three oxides of pure iron and may be regarded as solid solutions of chromium and oxygen in wüstite, magnetite or hematite. In spite of the evidence that pores and cracks play a major role in the kinetics of oxidation of these alloys, it is desirable to see to what extent the oxidation can be rationalized in terms of the Fe-Cr-O equilibria and information now available on diffusion in the three oxide structures. The most that can be expected is qualitative agreement with observed trends.

Fe-Cr-O Equilibria

Above 815 C, where a maximum occurs in the sigma phase alloy field, and below the solidus minimum in Fe-Cr at about 1500 C the ternary diagram should have the form shown in Figure 29. The γ alloy phase does not occur below 830 C or above 1400 C, but the only effect of its absence is to remove the dashed triangle and to convert all γ 's to α 's on Figure 29. The diagram substantially as given here seems to have been proposed first by Woodhouse and White.¹¹ Approximate tie lines in the S + R field were obtained earlier by Richards and White.¹² The diagram is similar to those given by Yearian et al.⁹ and by Edström.¹³ Seybolt¹⁴ has determined some additional equilibria which result in substantial changes in limiting compositions. Isobars in air have been obtained for Cr_2O_3 -Fe $_2\text{O}_3$ -FeO by Muan and Somya.¹⁵

Several things are clear from the nature of the equilibrium diagram. Wüstite is not stable in contact with an iron alloy containing appreciable amounts of dis-

solved chromium. The concentration of chromium necessary to stabilize the spinel phase in contact with alloy probably increases with increasing temperature. One oxidized specimen which initially contained 0.34 percent Cr showed a W layer as low as 818 C. 0.2 percent Cr alloys showed no W at or below 930 C after oxidation, but did have W at or above 1000 C. However, the fact that these alloys began to oxidize more rapidly than pure iron at temperatures as low as 850 C seems to require that W was present, because the other oxides are too slow growing to support such rates. A 2 percent Cr alloy had a W layer at 950 C. 5 percent Cr alloys gave no W as high as 1250 C. Because different oxidation times were employed and the distribution of chromium is not known in detail, quantitative equilibrium data cannot be obtained from these oxidation rate experiments.

Cr is taken into the spinel phase preferentially, according to the equilibrium diagram and according to studies of growing scales. If W forms only after S growth has depleted the alloy, in order for W to continue to grow either some Cr must dissolve in W or W must be in equilibrium with alloy containing a measurable concentration of Cr. No determination of Cr solubility in W has been reported. However, the fact that in the long run W grows more slowly on 0.2 and 0.34 percent Cr alloys than on pure Fe suggests that Cr partitions preferentially into the alloy phase under these conditions. With properly prepared samples it may be possible to check this point with electron microprobe x-ray analysis in spite of the low solute concentrations.

The fact that Cr partitions preferentially into the S layer from the alloy agrees with the analyses given in Figures 25 and 28, and also with analyses of total Cr content in scales.^{4,9} If the Cr-depleted surface is exposed to direct reaction with oxygen as a result of cracks passing through the scale, it is to be expected that the rate of oxidation might accelerate enormously.⁶ Any nickel in the alloy initially is likely to remain preferentially in the alloy phase regardless of whether the oxide growing next to the alloy is W, S or R.¹⁶ Therefore the consequences of breakaway are not likely to be so serious in a nickel-containing stainless steel as in a binary iron-chromium alloy, all other factors such as crack size, prior scale thickness, etc., being equal.

In order to make further use of the equilibria, it is necessary to know something about the ion mobilities in the ox-



Figure 16—Same specimen as Figure 14 at higher magnification. Subscale is clearly visible in the alloy layer. Etched in 4 percent nital, approximately 325X.

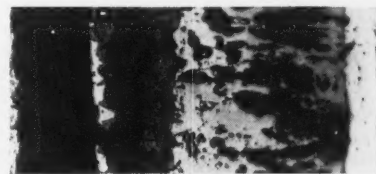


Figure 17—Same specimen as Figure 14 showing massive S growing on both sides of the W layer. Etched in 4 percent nital, approximately 50X.

ides, particularly in the spinel. From measurements made to date it is apparent that cation mobilities are generally lower in chromites than in ferrites,¹⁷ so that the overall rate of spinel growth is expected to be lower when the composition approaches more closely to FeCr_2O_4 . The studies by Yearian et al.^{9,4} on the distribution of cations in spinel layers on iron-chromium alloys indicate that the iron ions diffuse more rapidly leaving a concentration of chromium ions nearer the alloy side in agreement with the observations of Portevin et al.¹ The microstructures confirm this chromium accretion indirectly. In order for R to precipitate on the inner side of the S layer where the effective dissociation pressure is relatively low, chromium must be highly concentrated, so that R rich in chromium is the product. The analyses in Figure 25 show that Cr does not reach the outer part of the scale while Cr is depleted in the alloy. (The hematite layer should grow at about the same rate as it does on pure iron.)

The functions of chromium in the oxidation reaction then are the following: (1) Reduction in the rate of W growth

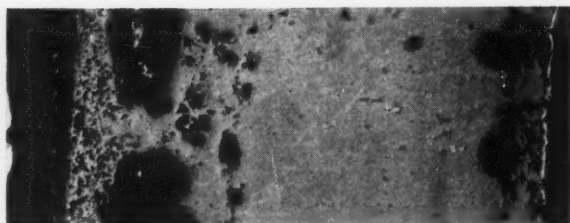


Figure 18—0.34 percent Cr alloy held 16 hours in hydrogen then oxidized 103 hours at 850 C. Unetched, approximately 75X.



Figure 19—0.34 percent Cr alloy held 17 hours in hydrogen then oxidized 27 hours at 900 C. Etched in 4 percent nital, approximately 160X.



Figure 20—Same specimen as Figure 19 at higher magnification, showing the unidentified inner layer and massive S growing into the W layer. Etched in 4 percent nital, approximately 370X.

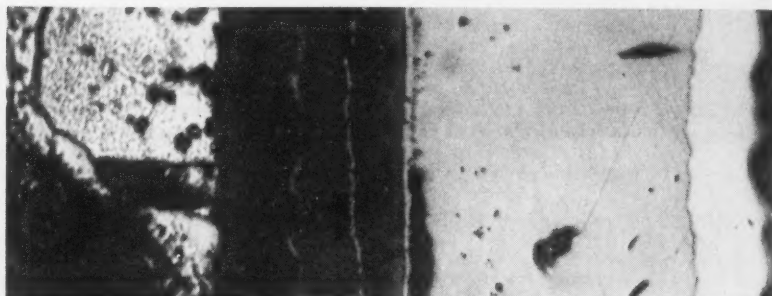


Figure 21—Two percent Cr alloy oxidized 3.4 hours at 960 C, showing S veins in the cracked W region. Etched in 5 percent nital, 200X.

by decreasing its range of stability and, if enough Cr is present, elimination of W as a product. This is the same kind of rate-reducing mechanism found for Ni in Fe,¹⁶ but much less Cr is needed. (2) Slowing the growth rate of S but not so much as of W for small Cr additions. (3) Elimination of S as a stable phase if the Cr content is high enough. Although Cr is taken preferentially into the oxide, when the oxides are slow growing, chromium depletion in the alloy near the interface must be reduced by interdiffusion of iron and chromium in the alloy phase. (4) When the Cr content is too high for S formation, or when S approaches FeCr_2O_4 in composition, the R layer may exhibit a modified growth rate as a result of increasing its Cr content. (The natural growth rate of pure Cr_2O_3 ¹⁸ is slower than that of Fe_2O_3 ¹⁹ at the same temperature. It can only be assumed that the mixtures grow at intermediate rates

at this time.) The thickness of R relative to the total scale becomes greater as the Cr content in the alloy increases.

Seybolt¹⁴ observed that above 13 percent Cr only R of nearly pure Cr_2O_3 composition is to be expected as an equilibrium product. The appearance of oxides rich in iron on alloys containing more than 13 percent Cr is a sign of mechanical failure in the scale.

Moreau and Benard²⁰ have oxidized 18 percent Cr alloys for long times in air at temperatures greater than 900 C. They find a great deal of W in the products and also observe extensive subscaling, where the internal oxide belongs to the R phase. On 2.6 percent Cr alloys W was observed above 600 C, and the oxidation rates were close to those of pure iron. These observations suggest that the alloy near the scale was severely depleted of dissolved Cr in the long oxidation period. Although the use of cylindrical samples may have contributed to substantially different cracking conditions than those found in flat samples, it seems likely that W was not one of the initial products but only appeared after Cr depletion occurred.

On the other hand the W observed in the 0.2 and 0.34 percent Cr alloys was probably there from the start since many 0.2 percent Cr alloys oxidized *initially* faster than pure iron, becoming slower only after a half hour in some cases. It is not clear why some 0.2 percent Cr samples show W and others do not. It seems unlikely that S nucleation is easier than W nucleation, but some explanation of this sort may be required.

Yearian et al⁹ found W derivatives for much higher initial Cr concentrations, but their commercial steels had a considerable amount of Mn. MnO forms a continuous series of nearly ideal solid solutions with FeO .²¹ Since MnO has a lower dissociation pressure than Cr_2O_3 , the presence of Mn may greatly extend the range of conditions under which W is stable in spite of the likelihood that Mn also increases the spinel stability. The spinel products were also found to contain high Mn concentrations.⁹

The non-reproducibility of the rate measurements, including the initiation of breakaway, appears to be an essential feature of the process. Since the structure sensitivity persists when all three oxide

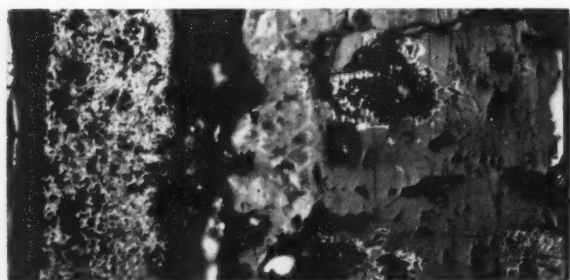


Figure 22—Five percent Cr alloy oxidized 3 days at 747 C, then 14 hours at 1250 C. Polarized light, approximately 80X.

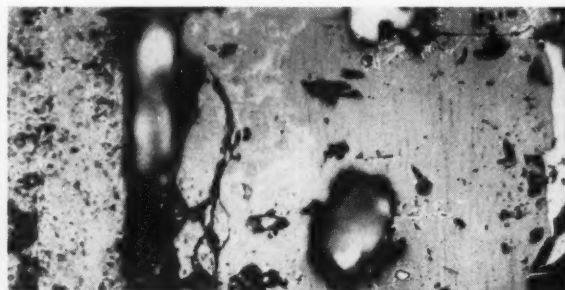


Figure 23—Same specimen as Figure 22. Polarized light, approximately 160X.



Figure 24—Same specimen as Figure 22, but showing mottled region only. Polarized light, 500X.

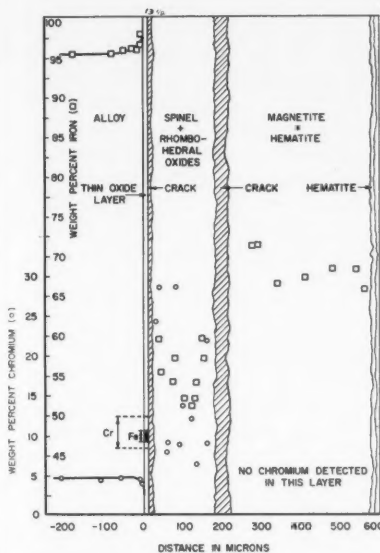


Figure 25—Metal ion concentrations in alloy and scale layers determined by microprobe analyses. The microstructure of the sample is indicated for comparison.

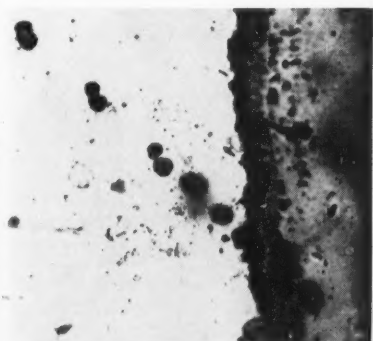


Figure 26—Five percent Cr alloy oxidized 12 hours at 850 C. Shows carbon deposition spots where the electron beam sampled the specimen. Unetched, approximately 660X.



Figure 27—Structure of the opposite face of the specimen in Figure 26. Unetched, 200X magnified to 280X.

phases are present in the sample, it appears that phase nucleation is not the dominant factor in giving the sporadic rate changes. Cracking seems to be the likely source of the irregularities, although the appearance of cracks has not been specifically correlated with breakaway in this study. Hornstra²² has shown that the Kronberg mechanism of strain rate dependent ductility²³ applies to spinels, as proposed earlier.²⁴ The alloying of magnetite should make it more brittle in several ways. Kronberg²³ showed that the glide of a dissociated dislocation through a complex structure, Al_2O_3 , in his example, tends to restore the lattice of one ion, leaving ions of the other sign in positions which are normally interstitial sites. To reduce the energy required for the dislocation to move on, the displaced ions must diffuse into proper positions. Among the spinels, reordering of the cations should be easiest in pure Fe_3O_4 , because a part of the ordering can occur by electron transfer, reducing the ion jump distances to a minimum.²⁵ When other cations are present, electron transfer may not be energetically as favorable. Furthermore, it has been shown that the ion diffusivities are lower in the substituted ferrites which have been studied,¹⁷ and the rates of ion diffusion to restore order should be reduced. In spite of the reduction in oxide growth rate with added Cr content, the brittleness may increase even more rapidly than the growth rate decreases. Pilling and Bedworth²⁶ found an effect of this sort with decreas-

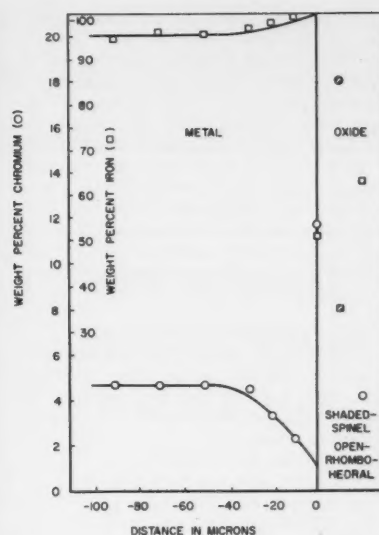


Figure 28—Metal ion concentration in the specimen, shown in Figure 26 as determined by microprobe analyses.

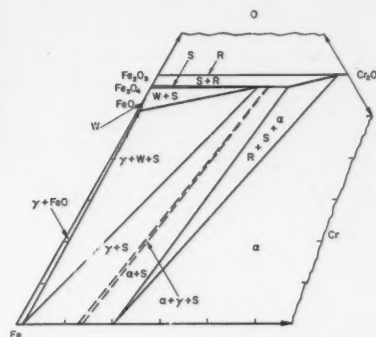


Figure 29—Isothermal triangle for the system Fe-Cr-O applicable with only small composition differences between 850 and 1450 C.

ing temperature for the oxidation of copper. Such behavior could account for the morphology of the oxide layers on the alloys.

Smeltzer²⁷ suggested that breakaway might be initiated when depletion of the alloying element in the alloy layer near the oxide leads to a phase change in the alloy. Such effects could contribute to these measurements only near 900 C for an alloy with 2 percent Cr or less and near 850 C for the 5 percent Cr alloys. Even then they could only be consistent with the results of this study if nucleation of the α - γ transformation occurred very erratically.

The effects of the hydrogen pretreatment are not understood. Perhaps the water vapor content was high enough to produce striations of the type observed by Moreau and Benard²⁸ and strongly modify the subsequent course of cracking and pore growth.

Fujii and Meussner²⁹ oxidized Fe-Cr alloys in helium-water vapor mixtures at 700, 900 and 1100 C. They found that W remained a major product up to 15 percent Cr content in the starting alloy. At 900 C the oxidation rates were somewhat slower than those of pure iron, but they

were much greater than the rate of oxidation of the same alloy in pure oxygen. The presence of hydrogen in the system has little effect on the growth rate of oxides on pure iron,¹⁰ therefore its large effect on these alloys is startling. In some manner hydrogen must increase the stability of W relative to S or accelerate the sub-scaling of the alloy so that internal precipitation of S keeps the chromium concentration in the outermost alloy layer low. Further investigation of the role of hydrogen in oxide transport processes and equilibria is clearly needed.

Acknowledgements

The authors are indebted to Mrs. Maria Borcsik who made some oxidation runs, took some of the X-ray diffraction patterns and prepared most of the metallographic cross-sections. They are also grateful to the International Nickel Company for the microanalyses reported here. This research was supported by the Air Force Office of Scientific Research (ARDC) under Contract Nos. AF 18 (600)-967 and 49(638)-533.

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DISCUSSION

Question by A. S. Couper, American Oil Co., Whiting, Indiana:

You mentioned that manganese would tend to stabilize the wüstite. Will you please elaborate on the possible mechanism?

Reply by C. E. Birchenall:

Evidence that manganese tends to stabilize wüstite during the oxidation of steels is indirect and of the two kinds cited. The thermodynamic evidence relates to the fact that MnO and FeO form a continuous series of solid solutions.⁹ Because MnO is a very stable oxide (that is, it has a low dissociation pressure relative to FeO and even low compared to Cr_2O_3) it is reasonable to suppose that the dissociation pressure of the mixed oxides is lower than that of FeO. Unlike nickel, and apparently chromium, there is no reason to believe that wüstite growing on a manganese-containing alloy does not contain manganese itself. The kinetic evidence is that Yearian and others⁹ who used manganese-containing steels found wüstite among the oxidation products at higher chromium concentrations than did the authors.

It should be reemphasized that it has not been directly demonstrated that wüstite is stabilized with respect to alloy and spinel. The pertinent section of the ternary equilibrium diagram would have to be worked out to make certain what is now a conjecture. Thus we believe that the presence or absence of wüstite depends only on its stability relative to alloy and spinel and not on kinetic considerations, because the cations in wüstite are sufficiently mobile to insure its growth.

Any discussion of this article not published above will appear in December, 1961 issue.



NATIONAL ASSOCIATION of CORROSION ENGINEERS



Driven Ground Rod Test Program

A Progress Report of NACE Task Group T-4A-3⁽¹⁾

On Methods and Materials for Grounding

**NACE TECHNICAL
COMMITTEE REPORT
Publication 61-5**

Introduction

THE INCREASED use of cement, plastic, wrapped pipe and even cast iron with neoprene-joint pipe systems for water mains may soon make useless the use of water pipes for grounding electrical systems. Many water systems install an insulating bushing between the house lines and meter or street mains.

Many of the water utility companies throughout the United States object to their piping systems being used as a ground for electrical systems.

The East Bay Municipal Utility District in Oakland, California, has a regulation: "Section 25. Prevention of Ground Wire Attachments.—The District is not responsible for providing an electrical ground through water service equipment. Accordingly, customers are cautioned not to attach any ground wiring to plumbing which is or may be connected to District service equipment. The District may hold the customer liable for any damage to its property resulting from a ground wire attachment."

At the October 1960 California Section Meeting of the American Water Works Association at Long Beach, there was extended discussion of the problem of connecting the electric neutral to water pipes. Two papers were presented entitled "Grounding of Electrical Services to Water Piping Systems" by Arthur G. Clark* and "Electrical Grounding Requirements Vs. Water Utility Practices" by Lee B. Hertzberg.**

Although there is no conclusive evidence that stray alternating currents from grounding on water piping systems materially affect electrolysis, electrolytic corrosion or galvanic corrosion, many of the small water companies are led to believe that some or all of their cor-

rosion problems are caused by the neutral grounding. It is quite possible that in the not too distant future the AWWA may go on record against such electrical grounding to their water pipes.

Work and Recommendations

The gas companies started a program some years ago to install an insulated meter swivel on the inlet side of the meter during periodic change. Their principal reason for doing this was to prevent an arc or spark when removing a gas meter from the service line.

The national, state and local codes and safety orders are all about the same in specifying that the grounding electrode shall be a continuous metallic underground cold water piping system where such piping system is available. Where a water system is not available, the grounding connections may be made to other local metallic underground piping systems or plate, pipe or rod electrodes.

It has been reported that the Southwestern Regional Conference of International Association of Electrical Inspectors at a recent meeting made a recommendation to panel five for inclusion in the 1962 code to drive individual ground rods at every service.

The City of San Francisco in 1961 will require a driven ground rod to be installed at each point where the neutral is connected to the cold water line.

The National Association of Corrosion Engineers through their Committee T-4A, "Effects of Electrical Grounding on Corrosion" have for several years been concerned with this grounding problem. Task Group T-4A-3, "Methods and Materials for Grounding," is starting a "Driven Ground Rod Test Program" to find a suitable material for ground rods, said material not to have the galvanic disadvantages of copper. This is a national program with test sites to be selected in various parts of the United States.

Copper Ground Rods

The material which has had the most

Abstract

Dangers involved in grounding of electrical systems to water pipes are reviewed briefly. Safety orders, municipal and state codes, technical society recommended practices, etc. are discussed in this connection. It was found that the City of San Francisco required a driven ground rod to be installed at each point where the neutral was connected to the water line.

A proposed driven ground rod test program is outlined briefly. Some 33 driven ground rods are scheduled to be examined after burial for one, three and seven year periods. Materials to be tested include mild steel, galvanized steel, copper clad steel, Ni-Resist and Type 302 stainless steel. Copper is not deemed to have the desired galvanic properties. 7.7

universal use is copper clad steel rods or copper tubing. Copper when connected to steel pipes, lead cable sheaths, etc., creates a bad galvanic situation. The copper receives cathodic protection from the other metals connected to it at their expense. The damage to steel when a small area of copper is connected to a large area of steel is not very great.

If the use of copper for ground rods for every electric service should become universal, all these copper ground rods connected together through the electric neutral would be a great hazard to steel connected to it for both the utility and the consumer.

Another factor to keep in mind is this: if all consumer grounding should be done with ground rods, who is going to maintain this grounding network? It is necessary to know what will happen when a large number of ground rods corrode away and the resistance to ground rises above the minimum of 25 ohms. The electric codes could be changed to require the utility to maintain all ground rods.

Other companies are also interested in this question. For example, the telephone companies need good grounds for their ringing circuits on party line telephones.

Occasionally a water softener is installed in water lines outside buildings and is connected with rubber hose fittings. This insulates the water pipe ground from the house water piping system.

The Water System of the Department

⁽¹⁾ Irwin C. Dietze, Dept. of Water and Power, City of Los Angeles, Los Angeles, Calif., Chairman.

* Arthur G. Clark, Acting Chief, Mechanical Bureau, Department of Building and Safety, City of Los Angeles.

** Lee B. Hertzberg, Supervising Mechanical and Electrical Engineer, East Bay Municipal Utility District, Oakland, California.

of Water and Power has received approval from the Board of Public Works to use plastic pipe for water services up to its water meter.

Summary

It can be readily seen that there are many persons concerned with this grounding problem and how to solve it. The "Driven Ground Rod Program" of the NACE is a step forward to acquire data for solving this problem. Both the utilities and the code regulatory bodies should welcome and help in this program.

NACE Committee Program on Driven Ground Rods

On June 17, 1960 the first definite proposal for a "Driven Ground Rod Program" was formulated and mailed to 52 committee members and others thought to be interested in this program. A meeting of Committee T-4A-3 was held September 8, 1960 in Los Angeles to consider suggestions received from the above mentioned letters and to revise the program. On October 7, 1960 a second committee meeting was held in San Francisco during the Western Regional Convention.

The second revised program dated September 29, 1960 was mailed October 21, 1960 to 90 people. The last paragraph of the letter asked for suggestions and stated that if addressees were interested in the program in any form whatsoever to please reply; otherwise their names would be removed from the mailing list.

The following types of $\frac{3}{8}$ -inch x 8 foot long ground rods were selected by the task group for inclusion in the program.

1. Mild Steel
2. Galvanized Steel
3. Copper Clad Steel
4. Ni-Resist
5. Stainless Steel, 302

Three complete sets totaling 33 driven ground rods will be buried at each site with the removal for examination of one complete set after one, three and seven years.

The task group received definite commitments from eight persons whose companies will sponsor a total of eleven test sites. Several other individuals are also in the process of obtaining sponsorship of a test site.

The International Nickel Company, Inc., agreed to furnish the Ni-Resist and stainless steel 302 rods. The Copperweld Steel Company agreed to furnish the copperweld steel rods, and the Tennessee Coal and Iron Co. the hot dipped galvanized steel rods. The balance of 12 mild steel rods will be furnished by each sponsor. It is estimated that the cost of these 12 rods will be less than \$45.00 for material.

In addition to the eight persons mentioned previously, 22 others answered the task group's inquiry of October 7, 1960 indicating their interest in the progress of the project but not participating.

The third and final revised "Driven Ground Rod Test Program," dated January 16, 1961, and showing details of plan, data sheets, etc., was mailed to all those sponsoring test sites and interested parties.

The task group is still looking for more sponsors in different parts of the country. Anyone desiring to participate in this program and sponsor a "Driven Ground Rod Test Program," is asked to contact the chairman of T-4A-3 as soon as possible.

**Any discussion of this article not published above
will appear in December, 1961 issue.**



NATIONAL ASSOCIATION of CORROSION ENGINEERS



Pipe-Type Cable Corrosion Protection Practices In the Utilities Industry

A Report by NACE Technical Unit T-4G
On Protection of Pipe-Type Cables⁽¹⁾

**NACE TECHNICAL
COMMITTEE REPORT**
Publication 61-6

Introduction

IN 1954, a report was published in *Corrosion* summarizing the corrosion control practices of 17 owners of pipe cable systems. These systems included 73 installations totalling 294.8 miles, installed between 1935 and 1953.

With the growing application of pipe cable systems and development of new corrosion mitigative methods, it became desirable in 1958 to make a survey of the pipe cables installed between 1953 and 1958. The results of the latter survey are reported herein.

This summary presents corrosion control practices on 301.2 circuit miles of pipe cable installed in the six-year period from 1953 to 1958. The information was supplied in response to a questionnaire distributed by Committee T-4G (formerly T-4B-4).

Eight companies which replied to the previous questionnaire also furnished information for the present survey. These companies include most of the large users of pipe-type cable in the United States.

⁽¹⁾ F. E. Kulman, Consolidated Edison Co. of New York, Inc., New York, N. Y., Chairman.

General Information

The following utilities submitted replies to the questionnaire: Baltimore Gas & Electric Company, Baltimore, Maryland; Boston Edison Company, Boston, Massachusetts; Central Power & Light Company, Corpus Christi, Texas; The Cleveland Electric Illuminating Co., Cleveland, Ohio; Commonwealth Edison Company, Chicago, Illinois; Consolidated Edison Company of N. Y., Inc., New York, New York; The Detroit Edison Company, Detroit, Michigan; Duquesne Light Company, Pittsburgh, Pennsylvania; Florida Power & Light Company, Miami, Florida; Long Island Lighting Company, Hicksville, New York; Memphis Light, Gas & Water Division, Memphis, Tennessee; Philadelphia Electric Company, Philadelphia, Pennsylvania; Potomac Electric Power Company, Washington, District of Columbia; Public Service Electric & Gas Company, Newark, New Jersey; Nikon Kokan K. K., Toyama, Japan; Sumitomo Chemical Ind., Ltd., Niihama-shi, Ehime-Ken, Japan; Yawata Iron & Steel Co., Ltd., Yawata-shi, Fukuoka-Ken, Japan.

Table 1 summarizes the general infor-

Abstract

A summary is made on corrosion control practices on 301.2 circuit miles of pipe cable for the period 1953 and 1958. High pressure oil cables made up 245.9 miles of the total and high pressure gas cables the remaining 55.3 miles. Information contained in this report was supplied by 17 utility companies in response to a questionnaire distributed by an NACE Technical Committee. Asphalt mastic pipe coating was used by 14 of the 17 companies. Three companies used coal tar enamel, two asphalt enamel and one epoxy coating.

Data given include number of circuits energized during year, approximate circuit miles and kilovolts, operating temperature design, average length between manholes, pressure medium (pipe miles), sizes of pipe used for pipe cables, type of external pipe coating used on buried pipe, type reinforcing and shielding used with enamel, coating thickness, precautions taken to prevent coating damage, material used to coat weld, and material used to coat cable joint sleeves in manholes. Also discussed were material used on non-buried sections, acceptable coating resistance, average soil resistivity, rate of coating deterioration, grounding and cathodic protection practices, performance of cathodic protection equipment, etc.

The resistor-rectifier method of cathodic protection was finding wider application among companies operating pipe cables. 7.7

mation on the pipe cable installations discussed in this report.

Of the 17 companies reporting instal-

TABLE 1—General Information On Pipe Cables Installed 1953-1958

Company	Name of Manufacturer*					No. Circuits Energized During Year								Approximate Circuit Miles and Kilovolts								Operating Temperature Design, °C				Average Length between Manholes, Feet	Pressure Medium, Pipe Miles	
	Gen	Ok	PD	FE	SE	'53	'54	'55	'56	'57	'58	All	26 Kv	66 Kv	69Kv	115 Kv	120 Kv	138 Kv	All	66-69 Kv Cables		115-138 Kv Cables		Oil	Gas			
																				Normal	Emergency	Normal	Emergency					
A	X	X	X					2	2	1	2	7			18.90			19.10	38.00	75	95	70	90	-	35.3	2.7		
B	X	X	X				6	2	4	10	1	5	28			20.84			29.92	50.76	75	95	70	90	2260	33.36	17.4	
C	X	X	X						1	1		2						24.50	24.50	-	-	-	55	60	2230	24.50	-	
D	X	X	X				1			4	1	1	7	2.50					55.90	58.40	-	-	70	100	2000	58.40	-	
E	X			X						2		4						15.04		15.04	-	-	70	70	2580	15.04	-	
F	X	X							2			2					9.0		9.0		-	-	70	90	2600	9.0	-	
G	X	X								1		1				2.20			2.20		-	-	-	-	-	2.2	-	
H		X										1	1					8.90	8.90	-	-	70	90	1570	8.9	-		
J	X						1	1	1	1	1	2	7					27.47	27.47	-	-	70	100	2100	-	27.47		
K	X	X									1	1	2			6.40			2.28	8.68	75	95	75	95	1361	8.68	-	
L			X				1					1				5.80				5.80	-	-	-	-	4800	-	5.8	
M			X					1					1				8.00			8.00	-	-	75	90	2200	8.0	-	
N	X	X	X				1		1	4	4	2	12			15.04			.24	15.28	75	95	70	90	2900	15.28	-	
P	X	X	X								3	1	4			27.29			27.29		75	95	-	-	2310	27.29	-	
Q				X						1		1	1		.20			.20		.20	75	90	-	-	-	-	0.20	
R				X							1		1		.20			.20		.20	80	80	-	-	-	-	0.20	
S				X						1		1	1		1.50			1.50		1.50	80	80	-	-	-	-	1.50	
Total	10	10	9	1	2		10	6	11	27	14	14	82	2.50	1.90	96.47	17.00	15.04	168.31	301.22						245.95	55.27	

*Gen-General Cable; Ok-Onokite; PD-Pheips Dodge; FE-Furnkama Electric; SE-Sumitomo Electric

lations of pipe-type cables, the manufacturers of their cables were General Cable Corporation, Okonite Company, and Phelps Dodge Copper Products Corporation in the United States and Furukawa Electric and Sumitomo Electric in Japan.

The 17 users reported a total of 301.2 circuit miles of pipe-type cable installed, with voltage distribution as follows:

Kv	26	66	69	115	120	138
Miles	2.5	1.9	96.5	17.0	15.1	168.3

TABLE 2 — Sizes of Pipe Used For Pipe Cables

WALL THICKNESS, INCHES	NUMBER OF COMPANIES INDICATED					
	4 3/8*	5 9/16*	6*	6 5/8*	7*	8 3/8*
0.177	1	-	-	-	-	-
0.250	-	1	3	4	1	6
0.258	-	5	-	-	-	-
0.277	-	-	-	-	-	2
0.280	-	-	-	2	-	-
0.322	-	-	-	-	-	2
0.375	-	-	1	-	-	-
0.406	-	-	-	-	-	1
0.432	-	-	-	1	-	-
0.500	-	-	-	-	-	2

*OD in inches

The pipe-type cables reported are of two types: high pressure oil cables and high pressure gas cables. The total mileage of high pressure oil cables is 245.9 miles, and of high pressure gas cables 55.3 miles.

The average length of pipe between manholes for the various installations ranged from 1,361 feet to 4,800 feet. The maximum length of pipe is limited by the allowable stresses in the power cable during pulling in operations and is maximized when the number of pipe bends is a minimum.

The sizes of pipe installed, and their wall thicknesses are shown in Table 2.

Pipe Coating

Asphalt mastic pipe coating was used by 14 of the 17 companies. Three companies used coal tar enamel, two asphalt enamel, and one company used epoxy coating. The coal tar enamel and asphalt enamel coatings were reinforced with fiber-glass. Asbestos felt was reported as an outer shielding material by two companies.

Fifteen companies used sand or sifted earth padding to protect the coating from

penetration by sharp objects. One company poured a concrete envelope around the pipe. One Japanese company used rock shield placed around the coating. Curved sheet metal plates were placed around the pipe by one user where the pipe was in close proximity to other structures.

Pipe welds in mastic coated pipes were coated with asphalt mastic by eleven users, and with asphalt enamel by four users. Asphalt enamel was used to coat the welds by the two companies using asphalt enamel pipe coating. Two of the three companies using coal tar coatings used the same material for coating the welded joints and one company used asphalt enamel.

Fourteen companies reported that they had not experienced deterioration of the pipe coating. Two companies reported deterioration of asphalt mastic coating. One company did not reply to the question on deterioration.

Thirteen companies reported specific values of minimum coating resistance (megohms per sq. ft. of pipe area) acceptable on initial tests of the coating. Eight of these gave 10 megohms per

TABLE 3—Pipe Coatings and Their Performance

Company	Type of External Pipe Coating Used on Buried Pipe	If Enamel, What Kind of Reinforcing and Shielding Used?	Nominal Thickness of Coating (Inch)	Precautions Taken to Prevent Coating Damage	Material Used to Coat Pipe Weld	Material Used to Coat Cable Joint Sleeves in Manholes
A	Asphalt mastic			Sand padding	Asphalt mastic or asphalt enamel	Black Paint
B	1. Asphalt mastic 2. Coal tar enamel	Fiberglass and asbestos felt	1/4 for asphalt mastic 3/32 for coal tar	Sand padding	1. Asphalt mastic 2. Coal tar enamel	Coal tar enamel
C	1. Asphalt mastic 2. Coal tar enamel	Fiberglass and asbestos felt	1/4 for asphalt mastic 3/32 for coal tar	Sand padding	1. Asphalt mastic 2. coal tar enamel	Grease and laminated wrapper
D	1. Asphalt mastic 2. Epoxy		1/2	Sand padding	Asphalt-enamel	Glass fabric sat. with asphalt
E	Asphalt mastic		1/2	Sand padding. Curved sheet metal plates are placed where pipe is in close proximity to other structures.	Asphalt mastic	Sand blast metal 1 Coat ACB tropical primer 2 Coats Sipes subway black
F	Asphalt mastic		7/16	Sand padding	Asphalt mastic	Sprayed metal & Sipes # 80 Paint
G	Asphalt mastic		1/2	Concrete envelope poured around pipe.	Asphalt mastic	Coal tar enamel on fiberglass
H	Asphalt mastic		1/2	Sand padding	Asphalt-enamel	Semi-quick drying phenolic red lead
J	Asphalt mastic		1/2	Sand padding	Asphalt mastic	Pipe is coated with asphalt mastic. Valves, gas tie, copper pipes, etc., coated and wrapped with No-Ox-Id.
K	Asphalt mastic		1/2	Sifted earth	Asphalt mastic	Cold applied tape coat
L	Asphalt mastic		1	Sand padding	Asphalt mastic	Asphalt mastic
M	Asphalt mastic		7/16	Sifted earth only where necessary	Asphalt mastic	Pipe coated with sprayed aluminum. Sump pumps are used to keep manholes relatively dry.
N	Asphalt mastic		1/2" for 8" Pipe 3/16" for 6" Pipe	Sand padding	Asphalt enamel or asphalt mastic	Sprayed metal and synthetic resins
P	Asphalt mastic		7/16	Sand padding	Asphalt enamel	Asphalt enamel
Q	Reinforced coal tar enamel	Asphalt impregnated glass fiber tape and jute yarn.	1/2	Sand padding	Asphalt enamel	(No joint in pipe line)
R	Reinforced asphalt enamel	Glass, jute and cotton fabric tape.	3/16	Sand padding	Asphalt enamel	(No joint in pipe line)
S	Reinforced asphalt enamel	Glass, jute and cotton fabric tape. No shielding.	3/16	Rock shield	Asphalt enamel	No joint

square foot as minimum acceptable resistance. Five other utilities reported respective individual values of 2.3, 16.95, 17, 17 and 30 megohms per square foot of pipe area, as the minimum acceptable. Two companies, using coal tar coating, reported individual values of 0.3 and 2.8 megohms per square foot of pipe area as the minimum acceptable for this material.

With respect to decreases of coating resistance with time, most respondents stated that they could not indicate the rate of depreciation of coating resistance. However, one company stated that the resistance of asphalt mastic decreased approximately 99 percent in five years. Another company, with a minimum acceptable resistance of 30 megohms per square foot, reported decreases to between 0.3 and 0.5 megohm per square foot in five years.

Other companies reported decreases in individual installations of 13 percent in one year and 50 percent in four years. No information was reported on decreases in asphalt or coal tar coatings.

Table 3 shows the replies to the question on coatings.

Grounding

Grounding the pipe is necessary from the standpoint of safety. The provisions for grounding may complicate the cathodic protection and vice versa. Numerous methods of achieving grounding and cathodic protection were reported by eleven companies.

Six companies connect the source end of the pipe directly to the station ground bus with a cable connection. Six companies ground the source end of the pipe, through a resistor of 0.003 to 0.004 ohm, to the ground bus and energize the resistor by a cathodic protection rectifier.

Three companies ground the source end of the pipe through a 2-volt storage cell.

Two companies do not ground the pipe directly but provide connection through low voltage spark gaps or cut-outs.

Cathodic Protection

Thirteen companies provide cathodic protection to their pipes and four do not. The type of cathodic protection applied is intimately related to the grounding method.

Four of the six companies which ground their pipe directly to the station ground bus do not use cathodic protection. The remaining two connect the pipe along its route to sacrificial anodes.

Five of the six companies which ground the pipe through a resistor, connect a rectifier across the resistor to build up a potential difference in the order of 0.4 volt between the pipe and the station ground. The sixth company connects a rectifier between the pipe and a buried anode at the station end of the line. Supplementing the protection provided by the station rectifier, two companies protect the pipe between the stations by a rectifier and buried anode and another company connects the pipe to buried magnesium anodes at each manhole.

The three companies which ground the pipe through a storage cell—use the cell as a source of cathodic protection current.

The two companies which provide low voltage spark gaps or cut outs in the ground connection, connect the pipe to sacrificial anodes along the pipeline. In addition, one of these companies connects its pipe to sacrificial anodes near the station ground rods.

TABLE 3 (Continued)

Material Used on Non-Buried Sections	Cases of Coating Deterioration Experienced	Acceptable Coating Resistance (megohms per sq. ft.)	Average Soil Resistivity (ohms-cm.)	Can You Indicate the Rate of Depreciation of Coating Resistance?
1 inch Hevi-cote on submarine crossing	No	10	Above 32,000	Coating resistance measurements are holding fairly well. One installation, however, decreased about 50% in four years.
Underwater sections are coated with reinforced and shielded coal tar enamel and a reinforced concrete covering. Sections in underwater tunnel are protected by paint or epoxy-coal tar coating.	Yes*	1. 17 2. 2.8	8,000 to 15,999	Our tests show asphalt mastic resistance decreases approx. 99% in 5 years.
Copper risers protected with pressure sensitive tape.	No	17	2,000 to 3,999	One asphalt mastic coated line checked after one year showed coating resistance had decreased 13%.
Asphalt mastic and cement underwater. Asphalt enamel on bridge.	No	10	Unknown	No
In tunnel pipe is insulated from tunnel attachments. Pipe is grit blasted; Subox Galvanox at point of attachment to concrete 1 Coat ACB Primer & 1 Coat Tropilite Topcoat. In stream crossing, Hevi-cote over asphalt mastic.	No	10	No data
In tunnel pipe protected by 2 Coats Sipes #80 paint.	No	10	Not known	Insufficient time in service.
Portion of pipe in channel was laid in trench and is protected by 1 inch Hevi-cote re-inforced concrete.	No	2.3	100 (Est.)	Unknown
Pipe on bridge protected by asphalt mastic.	No	16.95	Not available	No, additional measurements are required.
On Bridge—asphalt mastic underwater—Hevi-cote	No	10	4,000 to 7,999	Not sufficient data available due to fact that we have not been able to take lines out of service for tests.
Underwater—½ inch asphalt mastic with 1 inch reinforced cement.	No	10	0 to 1,999	Not to date
No non-buried sections	No	..	0 to 1,999	No
No non-buried sections	Yes**	10	4,000 to 7,999	No
Pipes on bridges and in tunnels are protected by paint coating.	...	30	2,000 to 32,000	Installations show coating resistance decreases asymptotically to between 0.3 and 0.5 megohm/sq. ft. in 5 years.
Underwater pipes are protected by 1 inch reinforced concrete over asphalt mastic.	No	10	4,000 to 7,999	No
No Experience	No	0.3	2,000 to 3,999	No
	No	No
Pipe installed in tunnel on porcelain rollers	No	No

*In tide-water manhole, with pipe at two volts negative potential to other structures, it was found that paints, PVC tapes, and Thiokol coatings failed. As a protective measure coal tar enamel has performed very well.

**Coating was damaged by gasoline and by magnolia roots. PVC tape was applied in gasoline areas. Galvanized sheet steel used in root areas.

There was a wide range of answers to the question of pipe-to-soil potential sought for. With respect to the eight companies using copper sulfate electrodes for earth contact, three companies state that minus 0.85 volt was their sought for potential, and five companies reported desired potentials of 0.7, 1.0, 1.0, 1.2 and 2.2 volts. With respect to the five companies using steel test electrodes, three looked for a pipe potential of minus 0.3 volt with respect to the test electrode, one for a minus 0.4 volt, and one for a 0.3 volt change in pipe potential. Of the four remaining companies, three did not reply to the question and the other replied "0.4 to 0.8 volt negative to soil."

With the view to reducing the anodic exposure of other underground structures resulting from the application of cathodic protection to the pipe-type cables, three companies make tests jointly with owners of structures in the vicinity of the pipe. Three companies maintain their coating at a high level of resistivity, one controls the level of cathodic protection, one provides for bond connections to other pipes, and one stated that the other structures are protected by magnesium anodes. Two companies stated that the pipe cable lines are installed in areas relatively free of underground structures, three companies make no provision for minimizing the anodic exposure of the

other underground structures. One company using cathodic protection did not reply to this question.

Performance of Cathodic Protection Equipment

Cathodic protection equipment may be subject to potential damage from faults in the pipe cables, induced voltages in the pipe, and momentary surges caused by lightning and by switching of the cables.

Only two of the seventeen companies had experienced pipe cable failures so far and both replied that no damage had been caused to the cathodic protection equipment installed in the ground connections.

TABLE 4—Grounding and Cathodic Protection Practices

Company	Insulating Material Between the Pipe and Terminal Equipment?	How is Pipe Grounded at Source End?	Is Pipe Connected Electrically En Route?	Is Pipe Grounded at End Remote from Source?	Insulating Joints in Cable Pipe?	Cathodic Protection Used?
A	Yes	Storage cell in connection to ground bus.	Normally not grounded. However, one short installation recently required sacrificial anodes.	Yes. Storage cell in connection to ground bus.	No	Yes
B	Yes	1. 0.004 ohm resistor in connection to ground bus. 2. Secondary cell in connection to ground bus.	To cathodic protection rectifier at submarine crossing. To pipe of adjacent pipe cable in same manhole	Yes. On each feeder, the grounding at the remote end is the same as at the source end.	No	Yes
C	Yes	Westinghouse oxide film cut-out in connection to ground bus. Pipe connected to a magnesium anode buried below floor of each manhole.	To pipe of adjacent pipe cable in same manhole and to sacrificial anode at each manhole. To cathodic protection rectifier being installed (1957).	Yes. Through sacrificial anodes and to station ground bus through voltage breakdown gap.		
D	Yes	Resistor in connection to ground bus.	To sacrificial anodes at one location only.	Yes. Resistor in connection to ground bus.	No	Yes
E	Yes	0.003 ohm resistor in connection to ground bus.	Not grounded	Yes. Both ends grounded through resistors to ground bus.	No	Yes
F	Yes	Through resistor through which cathodic protection current flows.	Yes. Through spark gap.	No	Yes
G	Yes	Pipe connected to sacrificial anodes near station ground rods.	To sacrificial anodes.	Yes. Pipe connected to magnesium anodes.	No	Yes
H	Yes	Final installation to include resistor in connection to ground bus with cathodic protection rectifier between pipe and graphite anodes.	Not grounded.	Yes. The grounding at the remote end is the same as at the source end.	No	Yes
J	Yes	Cable connection to ground bus by means of four parallel 1/0 rubber insulated cables.	To sacrificial anodes at two specific locations.	Yes. Pipe is grounded by means of four parallel 1/0 rubber insulated cables to station ground mass at each end of line.*	No	Yes
K	Yes	Spark gap in connection to ground bus. Pipe connected to sacrificial anodes near station ground rods.	To pipe of adjacent pipe cable in same manholes. To sacrificial anodes.	Yes. Through spark gap connected to ground bus.	No	Yes
L	Yes	Storage cell in connection to ground bus.	To sacrificial anodes.	Yes. Storage cell in connection to ground bus.	No	Yes
M	Yes	Resistor in connection to ground bus with rectifier connected across resistor for partial cathodic protection.	To sacrificial magnesium anodes.	Yes. Same as at source end.	No	Yes
N	Yes	Cable connection to ground bus.	Ground rods in manholes. Drainage connections to railway negative bus.	Yes. Same as at source end.	No	Yes
P	Yes	Cable connection to ground bus.	Not grounded.	Yes. Cable connection to ground bus.	No	No
Q	No	Cable connection to ground bus.	To copper or copper-weld ground rods.	Yes. Cable connection to ground bus.	No	No
R	No	Cable connection to ground bus.	To copper or copper-weld ground rods.	No. (Earth plate is consumed by the stray current).	No	No
S	No	Cable connection to ground bus.	To copper or copper-weld ground rods.	Yes	No	No

* One line has a polarization cell inserted between the pipe and ground connection at each end.

Four companies stated that they had observed the presence of continuing induced voltages or currents in the pipe. One company stated that 6 volts a-c was measured, but that the source of induction was not determined. A second company traced the induction to a traction system and improved the condition by installing drainage bonds, and another company stated that an electrolytic plant operated near the pipe line and mitigation was obtained by omitting the ground plate. The fourth company did not identify the nature or cause of the inductions.

Five companies reported transient pipe voltages or currents. Two companies reported that spark gaps at the remote end of the pipe flashed over (the source end

was grounded through low resistance cathodic protection equipment). One of these companies identified the cause as switching surges and corrected the condition by installing a grounding resistor and cathodic protection rectifier at the remote end. The other company replaced the spark gaps with thyrister protectors. One company, whose lines were protected by magnesium anodes buried below its manholes reported that the oxide film cut-out in the grounding connection became fused and thus grounded the pipe. The fusing was eliminated by connecting a one-ohm resistor between the pipe and the station ground. A fourth company, whose former method of grounding was through spark gaps, re-

moved the gaps and installed the resistor-rectifier method of cathodic protection at both ends of the pipe. The fifth company found that, notwithstanding the resistor-rectifier method of protection at both ends of the pipe, switching surges caused flashover of insulating flanges in the oil lines connected to the cable pipe and that corrective measures were being investigated.

Discussion

A comparison of the replies to the current questionnaire and the previous one suggests that several trends are evident in the corrosion protection of pipe cables.

A wider variety of coatings is now available with the introduction of the

TABLE 4 (Continued)

What Type of Cathodic Protection?	Value of Pipe-to-Soil Potential Sought for	Oil or Gas Supply Lines Insulated From The Cable Pipe?	Provisions Made to Minimize Anodic Exposures of Other Underground Structures Resulting From the Application of Cathodic Protection
1. Secondary cell in ground cable connection. 2. Sacrificial anodes along pipe line. 3. Drainage connection to railway system.	—1.0 volt to CuSO_4 electrode over pipe.	Yes	Maintenance of high resistance coating on pipe. Control of cathodic protection level.
1. Current—energized resistor in ground cable connection. 2. Secondary cell in ground cable connection. 3. Cathodic protection rectifier between pipe and buried anode at submarine crossing.	—0.3 volt to clean steel electrode in earth.	Yes	Provision and maintenance of high resistance coating on pipe. Tests with other utilities, using temporary installation of cathodic protection.
1. Sacrificial anodes along pipe line. 2. Rectifier being installed between pipes and negative returns of street railway system (1957).	—1.0 volt to remote CuSO_4 half cell.	No	None at the present time.
1. Cathodic protection rectifier between pipe and ground bus. 2. Cathodic protection rectifier between pipe and buried anode. 3. Sacrificial anodes along pipe line but not tied in as yet except as noted.	—0.3 volt	Yes	None required.
Current—energized resistor in ground cable connection at both terminals.	0.3 volt change	Yes	Crossings checked but no damaging effects on other structures by cable lines were found.
Current—energized resistor in ground cable connection.	—0.85 volt referred to CuSO_4 half cell.	No	High resistance asphalt mastic coating limits cathodic protection current to 100 to 200 milliamperes.
Sacrificial anodes along pipe line.	Measured —1.23 to —1.40 volts referred to CuSO_4 electrode.	Yes	All other pipes in crossing have individual magnesium anodes.
Cathodic protection rectifier between pipe and buried anode. Final connection not completed.	—0.85 volt to CuSO_4 half cell.	No. Pumping plant and storage tanks are insulated from supply lines.	Two cathodic protection systems along line and plan to have capacity available for ties if requested.
Sacrificial anodes at underwater crossings on two of our lines.	—0.85 volt to CuSO_4 half cell.	Yes	Portion of pipe under protection in area relatively free from other underground structures.
Sacrificial anodes along pipe line.	0.4 to 0.8 volt negative to soil.	Yes	As anodes are connected, Pittsburgh Corrosion Committee is advised and joint tests are made.
Storage cell in ground cable connection.	2.2 volts	Yes	Pipeline is isolated from others.
Current—energized resistor in ground cable connection at each end of each circuit. Two magnesium anodes at each manhole.	—0.7 volt referred to CuSO_4 half cell.	No. Oil lines are insulated from the oil pressure control devices.	None
Sacrificial anodes are connected to two pipes.	—0.3 volt	Yes
.....	—0.4 volt	Yes
.....			
.....			
.....			
.....			

TABLE 5—Performance of Cathodic Protection Equipment

Company	Have You Experienced Pipe Cable Failures?	If so, Was the Functioning of The Cathodic Protection Impaired?	Have You Experienced Continuing Induced Pipe Voltages or Currents While The Pipe Cables were Operating Normally?	What Was the Source of The Induction and How Was the Condition Improved?	Have You Experienced Effects of Transient Pipe Voltages or Currents While the Pipe Cable Were Operating Normally (e.g. Lightning, Switching Surges)?
A	Yes	No	Yes	No
B	Yes	No	No	Yes. When feeder 11001 (now 18001) was constructed in 1949 a spark gap was installed for cathodic protection purposes in the ground connection. The gap was installed in series with the ground connection between the pipe and the station ground bus. Several unexplained breakdowns of the gap occurred until staged switching tests showed that switching surges broke down the gap. The condition was corrected by replacing the gap with a 2 volt battery (later a resistor and rectifier) in the grounding connection.
C	No	...	Yes	A maximum of 6 volts a-c has been measured on the longest line. The source has not been definitely determined. No corrective measures have been taken.	Yes. Induced voltage caused the oxide film cutouts to fuse and ground the lines. At two locations a one ohm resistor was placed between the pipe and station ground.
D	No	...	No	Yes. On the 1953 line the original installation consisted of spark gaps in connection to the ground bus. These gaps were blowing frequently. The gaps were removed and the pipe tied to ground through a resistor with a rectifier energizing it.
E	No	...	No	No
F	No	...	No	Yes. Discharge of spark gap occurred at remote end. Spark gaps replaced with thyristor protectors in June of 1957 due to continual freezing together of spark gap contacts.
G	No	...	No	No
H	No	...	No	No
J	No. However, corrosion caused pipe failure in manhole under a potential wire connection.	No	No	No
K	No	...	No	No
L	No	No	No	No
M	No	...	No	Yes. During switching surges flashover occurred at insulating flanges with no apparent damage. Corrective measures are being investigated.
N	Yes	...	Yes	Traction system was source of induction. Condition was improved by drainage bonds.
P	No	...	No	No
Q	No	...	No	No
R	No	...	Yes	There is an electrolytic plant near the pipe line. Only the source end of the pipe is connected to the earth plate.	No
S	No	...	No	No

coal tar enamels possessing relatively high softening points (e.g., "hot line" enamel) and epoxy coatings introduced during the period covered by the survey. Little data are available on the performance qualities of the newer materials for pipe cables. However, one company reported that it had installed substantial mileage of coal tar enamel pipe (54 miles) in 1958. These installations were not included in the current report since they were energized in 1959. Future installations of pipe cable will be designed to carry greater amounts of energy and

coating performance should be equal to the future requirements.

The resistor-rectifier method of cathodic protection was introduced during the period covered by the report and is finding wider application among the companies operating pipe cables.

There have been relatively few failures of pipe cables. (The few failures which were reported were not necessarily caused by corrosion.) Thus the merits of the various cathodic protection schemes are yet to be evaluated from the stand-

point of withstanding damaging fault currents.

Additional study appears to be indicated for the problems of induced voltages in the pipes and the effects of switching surges in breaking down spark gaps in the grounding connections, and insulating joints in oil lines connected to the cable pipe.

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NACE Publication 54-3. Pipe-Type Cable Corrosion Protection Practices in the Utilities Industry. *Corrosion*, 10, No. 9, 299-313 (1954) Sep.

Magnesium Anodes for the Cathodic Protection of Naval Vessels*

By L. J. WALDRON and M. H. PETERSON

Introduction

THE U.S. Navy some eight years ago started cathodic protection of destroyer-type vessels with magnesium anodes. Various types of block magnesium anodes containing steel supporting straps were either bolted or welded to the bilge keel or hull plating. Both controlled and uncontrolled systems were used. After one or two years of operation, the vessels were docked and the hulls and anodes inspected. A general summary of the performance of cathodic protection obtained on these vessels after various periods of operation follows:¹

1. Cathodic protection was judged to be 90 to 100 percent effective in eliminating corrosion of the underwater hull. The degree of protection for the rudders, shafts, and struts was slightly lower, about 85 to 95 percent. In general, the protection afforded during the second year of operations, though still effective, was slightly less than that obtained during the first year.

2. The vinyl paint peeled at various areas of the keel and hull close to the magnesium anodes. Bare metal, free of rust, showed where the paint had peeled. Much of the coating was missing from the rudders, rudder skeg, shaft and struts. Light superficial surface rust was noted on the bare metal of these exposed areas. Except for a few isolated areas, no pitting was observed.

3. Most of the anodes were 60 to 85 percent consumed in a one-year period of operation. This deterioration was much greater than anticipated and was attributed to an excessive loss of current from the anodes to adjacent steel parts of the anode assembly and bare areas of the hull. Improved anode designs and installations are necessary to ensure a 24 to 30 month life.

4. The current required to maintain a given potential (0.75-0.85 volts to Ag-AgCl) increased with time during the first year, probably as a result of the exposure of increasing areas of bare metal from deterioration of the paint. At the end of the first year this was 2 to 4 times as much as at the start. The current requirements were essentially constant during the second year and indicated that comparatively little additional deterioration of the paint occurred.

5. Little difference due to velocity was noted in the current requirements once the vessel was underway, but approximately twice as much current was required to maintain a given potential when the ship was moving as when it



Waldron



Peterson

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was stationary. Grounding the vessels to piers or adjacent ships decreased the potential.

6. The potential determined by reference electrodes over the propellers was always 50 to 70 millivolts more cathodic than the average potential at various locations on the hull.

7. No evidence was obtained that cathodic protection either inhibited fouling or was interfering with the antifouling properties of the paint.

Objectives of Cathodic Protection

As a result of these preliminary experiences, the objective of cathodic protection of naval vessels can be set forth as:

1. The cathodic protection system should provide 24 to 30 months of freedom from corrosion of all underwater parts of the vessel.

2. It should be relatively inexpensive to install.

3. Operation of the system should not cause excessive deterioration of the paint.

4. It should require little or no maintenance. No attention should be required of ship's personnel.

Magnesium Anodes for Cathodic Protection

Magnesium has proven to be a dependable galvanic anode. Magnesium

Abstract

The paper describes the results of a cooperative investigation with the Bureau of Ships on the performance of magnesium anodes in the cathodic protection of naval vessels. Coated magnesium anodes were applied in 1958 to three destroyers and their performance evaluated after two years of operation. By attaching additional coated anodes to the number of uncoated anodes required for initial protection, and having divers remove some of the coatings from time to time (when indicated by potential measurements), a service life of three years is easily obtainable from one installation. All the vessels were docked after two years and their hulls found to be in excellent condition with little or no corrosion. 5.2.2, 8.9.5

anodes will deliver current until almost exhausted. Their current output is only slightly reduced with time since they do not tend to film over. For the protection of steel in seawater, their driving voltage is on the high side which will overprotect and waste current to adjacent bare areas. For this reason, all bare metal should be kept at a minimum.

The fact that appreciably more current will be required after a years' operation of the vessels suggests that any cathodic protection system should be able to increase its current output correspondingly. A conventional system with a fixed number of operating magnesium anodes will not increase the current output with time; in fact, if anything, its current output will decrease. The most practical way of accomplishing an increase in current is to add additional anodes after the first years' operation. This is difficult to do without docking which is a relatively expensive operation. The suggestion has been made that anodes be renewed underwater by divers without docking the vessel. While it may be possible to do this, it would be carried out with difficulty and might prove expensive.

Another difficulty in using a fixed number of uncoated anodes is that their number should be such as to supply a given current for some average condition of the hull. This means that an excess of current will be provided when the paint coating is fairly intact and new and that insufficient current will be furnished when the coating is old and badly deteriorated. Excessive current may hasten the deterioration of paint while an insufficient amount may result in corrosion of the hull.

The introduction some years ago of plastic-coated magnesium anodes as a means of controlling the current from these anodes suggested that by installing an excess of fully coated anodes at the original installation, and then having divers uncover additional anodes as they are needed, this might provide a system answering the specifications previously discussed.

Such systems were discussed with the Bureau of Ships and the leading suppli-

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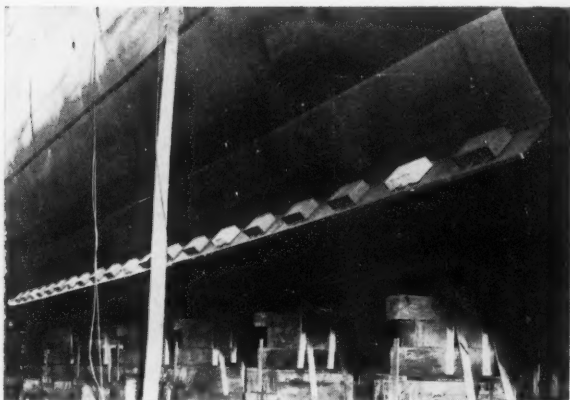


Figure 1—Original arrangement of coated magnesium anodes on one bilge keel.



Figure 2—Appearance of strut after 16 months' service.

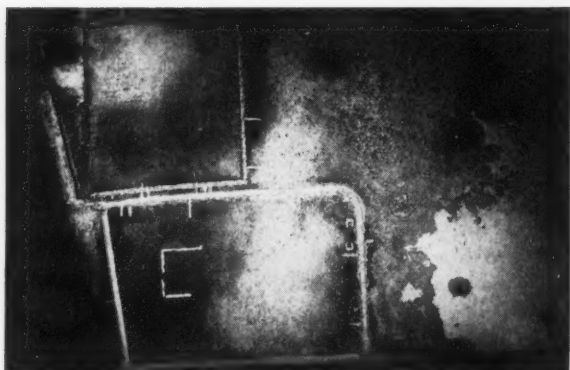


Figure 3—Appearance of rudder after 16 months' service.

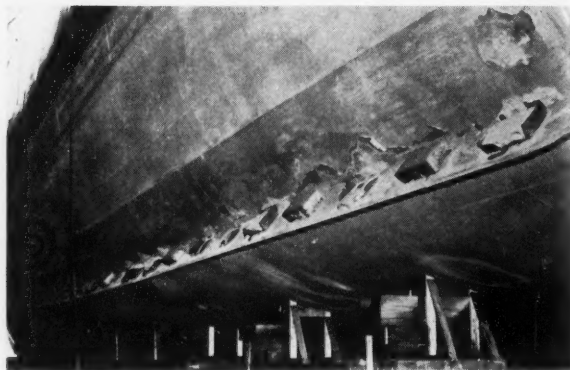


Figure 4—Appearance of anodes after 16 months.

ers of magnesium anodes with the result that three destroyers were outfitted in 1958 with experimental installations. The work was a cooperative project between the Bureau of Ships and the U.S. Naval Research Laboratory.

Installation Details

The three vessels selected for these experimental anode tests were:

USS Cecil	DDR-835
USS Stickell	DDR-888
USS O'Hare	DDR-889

The anodes were 9 inches wide, 18 inches long, and 4 inches thick and weighed approximately 44 pounds. Each anode contained two cast-in steel supporting straps. All anode surfaces and a portion of each supporting strap were coated by the manufacturers with a 3/16 inch plastic coating. The anode surface was not pretreated prior to application of the coating so that little or no adhesion existed between anode and coating. Twenty anodes were attached in a line on each keel about 18 inches apart between frames 100 and 130. They were secured by welding the supporting straps to the bilge keel. An extra thickness (25 mils) of vinyl coating extended on the hull 3 feet above the bilge keel. This coating also extended

under the bilge keel to the hull. The top plastic coating was stripped from selected anodes on both keels of each destroyer prior to undocking.

Figure 1 shows the arrangement of anodes on one of the vessels. The number of anodes uncovered originally on each vessel was as follows:

USS Cecil	10 each side
USS Stickell	7 each side
USS O'Hare	5 each side

After the vessels had been in service approximately one year, divers uncovered additional anodes as follows:

USS Cecil	10 each side
USS Stickell	8 each side
USS O'Hare	5 each side

The three vessels undocked the first part of November 1958 and were in active service until docked on 18 February 1960 after approximately 16 months.

Potential Measurements

After the three vessels had been in service for approximately one year, potential surveys were made of each hull while the ships were tied to piers at the Norfolk, Virginia Naval Operating Base. For these measurements, a silver-silver chloride reference electrode was immersed to a depth of 5 feet and as close

as possible to the hull at selected positions around the periphery of the deck. Approximately 24 positions were selected at intervals of every 20 frames (30 feet). A high resistance (200,000 ohms per volt) voltmeter was used. Similar measurements were again made in February 1960, a few days prior to the docking of the three vessels.

Typical potential data obtained from one of the vessels is shown in Table 1 with a general summary of the three vessels in Table 2.

The observed potentials were lowest at the stern undoubtedly due to the presence of the bronze propellers. Note that the potentials between frames 100 and 140 were nearly one volt or over. The potential readings at these locations were affected by the magnesium anodes attached to each bilge keel between frames 100 and 140. Consequently, the observed potentials here are not representative of those of the steel hull.

The potentials reported in these two tables were obtained while the vessels were stationary. They will drop when the vessels are underway. The exact decrease in potentials under these conditions is unknown since no means were provided for making measurements under moving conditions. The uncovering of additional anodes in November 1959, in general,



Figure 5—Appearance of an individual anode after 16 months, showing loss of contact with the steel supporting straps.

TABLE 1—Potentials (to Ag-AgCl) Obtained on the USS Cecil

Frame	STARBOARD, VOLTS		PORT, VOLTS	
	Oct. 1959	Feb. 1960	Oct. 1959	Feb. 1960
10.....	0.82	0.92	0.82	0.88
20.....	0.82	0.91	0.82	0.88
40.....	0.82	0.91	0.83	0.89
60.....	0.81	0.92	0.84	0.90
80.....	0.86	0.93	0.86	0.92
100.....	1.05	1.05	0.95	1.02
120.....	1.15	1.19	0.97	1.05
140.....	1.00	1.10	0.89	0.98
160.....	0.85	0.93	0.85	0.90
180.....	0.77	0.87	0.79	0.86
200.....	0.71	0.82	0.73	0.83
Stern.....	0.70	0.82	0.71	0.82
No. of anodes exposed.....	10	20	10	20

TABLE 2—Average Potentials Obtained on Three Vessels

Vessel	No. Exposed Anodes	Date	VOLTS TO Ag-AgCl		
			Bow	Midship	Stern
Cecil.....	20	15 Oct. 1959	0.82	0.85	0.72
	40	17 Feb. 1960	0.90	0.92	0.83
Stickell.....	14	15 Oct. 1959	0.72	0.81	0.72
	30	17 Feb. 1960	0.90	0.81	0.81
O'Hare.....	10	15 Oct. 1959	0.78	0.79	0.70
	20	17 Feb. 1960	0.74	0.79	0.75

had the effect of increasing the potentials of the steel hulls.

Inspection of Hulls—February 1960

After 16 months of operation, the hulls of all three vessels were nearly free from corrosion and could be considered excellent from this standpoint. A few iso-

lated spots of bare metal were covered with light superficial rust. The rudders and struts had lost a portion of their protective coating with subsequent exposure of bare metal. Except for one area on the rudder skeg of one ship, all exposed metal on these parts was free of rust and pitting. The coating was fairly intact on

all shafts with little or no rusting. All openings were relatively clean and free of corrosion. The paint on all three vessels was in fairly excellent condition. Some deterioration of the paint was noted on the hull just above the anodes. Figures 2 and 3 show typical areas of the struts and rudders with considerable exposed bare metal free from corrosion.

Performance of Anodes

Figure 4 shows the anode array on the bilge keel of one of the destroyers after 16 months' service. All anodes that had been uncovered throughout this period deteriorated to the point where little or no magnesium remained on the supporting straps. These particular anodes can be said to have a maximum life of 16 months.

Figure 5 is a close-up showing failure of a typical anode. After the bare metal of the strap is exposed, rapid deterioration of the magnesium occurs with loss of contact with the supports and eventual loss of the anode. The anodes uncovered by divers in November 1959 were 30 percent deteriorated in 4 months. The plastic coating of many of the anodes appeared to become brittle with age and to break off from the force of moving through the water. A coating that would adhere to the sides of the anode and the supporting straps should increase the life of the anode. Also eliminating the supporting straps and securing the anode by bolts or studs through the bilge keel should also increase the life of the anode.

Summary And Conclusions

1. By using a coated magnesium anode, a maintenance-free cathodic protection system can be installed on Naval vessels up to and including destroyer sizes that will provide 24-30 months of adequate protection to all underwater parts from corrosion.
2. More efficient anodes could be provided by eliminating the cast-in supporting straps and substituting a stud or bolt-type of attachment. The use of an adherent type of coating could also increase the efficiency of the anodes.

Acknowledgment

The cooperation of the officers and men of the three ships concerned in this work is gratefully acknowledged.

NOTE: The opinions expressed in this paper are those of the authors and not necessarily those of the U. S. Naval Research Laboratory or of the Navy Department.

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Any discussion of this article not published above will appear in December, 1961 issue.



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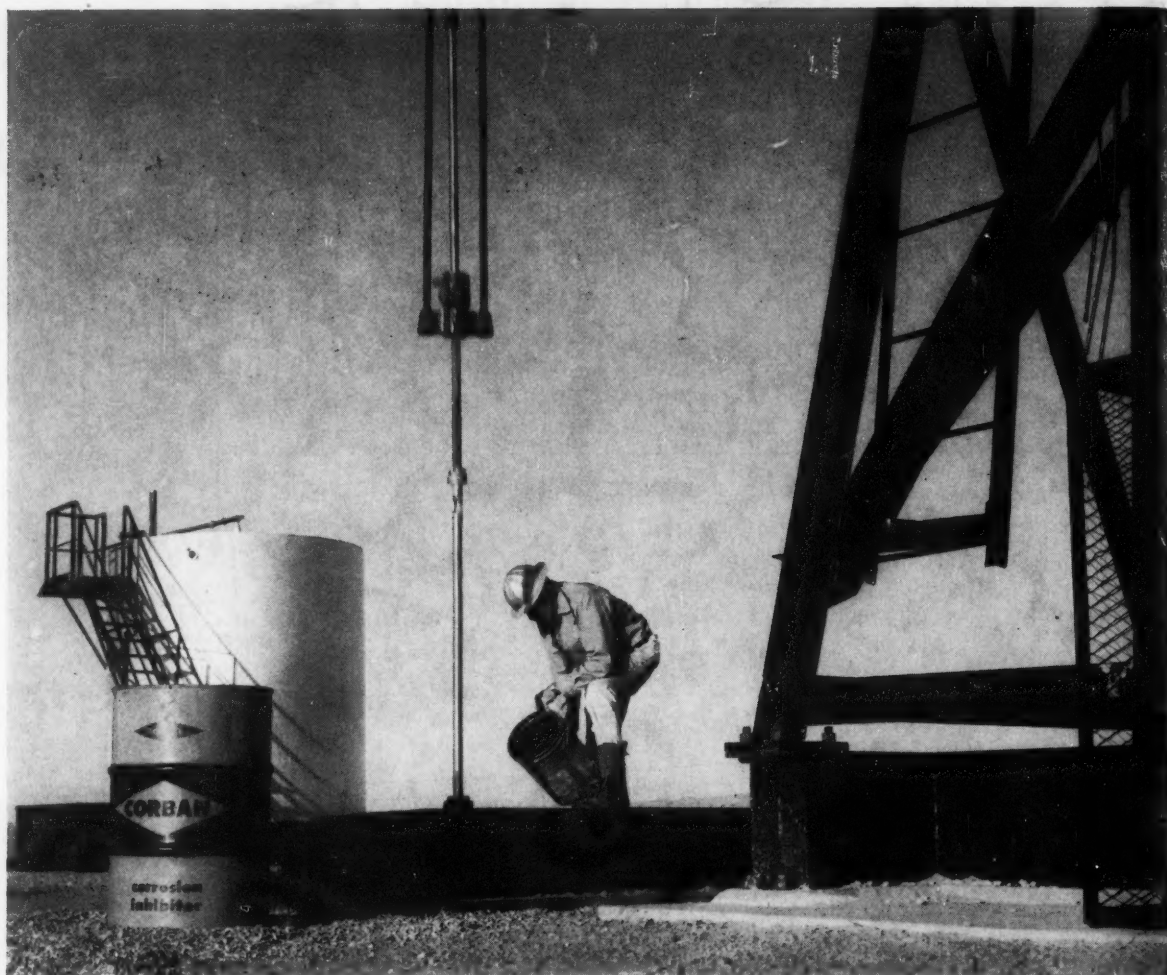
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